PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2003-282986
(43)Date of publication of application : 03.10.2003

(51)Int.Cl. H01L 41/09
C04B 35/46
C30B 29/32
H01L 41/187
H01L 41/22

(21)Application number : 2002-083702
(22)Date of filing : 25.03.2002

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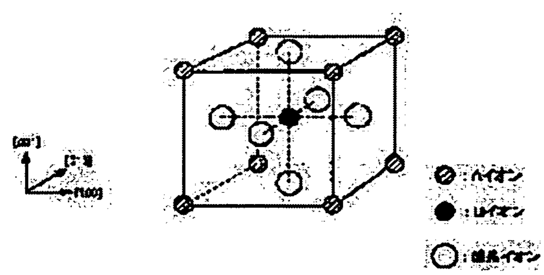
(54) DOMAIN CONTROL PIEZOELECTRIC SINGLE CRYSTAL ELEMENT AND ITS MANUFACTURING METHOD

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a domain control piezoelectric single crystal element wherein lateral direction vibration mode (k31) is used in which k31 is used effectively in the state that d33 is at least 800 pC/N when k33 is at least 80%, and -d31 is at least 1200 pC/N when k31 is at least 70%, and to provide a domain control piezoelectric single crystal element using a longitudinal vibration mode (k33) of high efficiency and high performance wherein spurious or the like is not present in a band width in which k33 vibration mode is used in the condition that d33 is at least 800 pC/N when k33 is at least 80%, and k31 is at most 30%.



SOLUTION: As a polarization condition in a thickness direction of a piezoelectric single crystal element, a DC electric field of 400-500 V/mm is applied for at most two hours in a temperature range of 20-200°C, or cooling is performed while an electric field is applied (electric field cooling). As a pre-stage of the above process, an electric field is applied to a direction perpendicular to a polarization direction (electric field application), or temperature is increased and decreased by setting a rhomboidal- tetragonal phase boundary temperature or a tetragonal-cubic phase boundary

temperature as a center temperature, or temperature is increased and decreased between different two temperatures in a cubic system temperature region (thermal treatment), or these temperature operations are used together.

LEGAL STATUS

[Date of request for examination]	16.09.2004
[Date of sending the examiner's decision of rejection]	
[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]	
[Date of final disposal for application]	
[Patent number]	
[Date of registration]	
[Number of appeal against examiner's decision of rejection]	
[Date of requesting appeal against examiner's decision of rejection]	
[Date of extinction of right]	

CLAIMS

[Claim(s)]
[Claim 1] In the piezo-electric single crystal ingredient which is electromechanical coupling coefficient k33 >=80% of the lengthwise direction oscillation mode of the direction of polarization, and has piezo-electric distorted constant d33 >=800 pC/N electromechanical coupling coefficient k31 >=70% of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization -- and It has piezo-electric distorted constant-d31 >=1200 pC/N. And the domain control piezo-electricity single crystal component characterized by being value fc31 <=650 Hz-m of the resonance frequency (fr) of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization about k31, and the frequency constant (fc31=fr-L) which is the product of the oscillating lay length (L) of a component.
[Claim 2] In the piezo-electric single crystal ingredient which is electromechanical coupling coefficient k33 >=80% of the lengthwise direction oscillation mode of the direction of polarization, and has piezo-electric distorted constant d33 >=800 pC/N electromechanical coupling coefficient k31 <=30% of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization -- and It has piezo-electric distorted constant-d31 <=300 pC/N. And the domain control piezo-electricity single crystal component characterized by being value fc31 >=800 Hz-m of the resonance frequency (fr) of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization about k31, and the frequency constant (fc31=fr-L) which is the product of the oscillating lay length (L) of a component.
[Claim 3] The domain control piezo electric crystal single crystal component according to claim 1 or 2 to which a piezo-electric single crystal ingredient is characterized by being following (a) or (b).
(a) It is the solid solution which consists of X-Pb(A1, A2, --, B1, B-2--) O3+(1-X) PbTiO3 (0< X<1). 1 or two or more elements which were chosen from the group which A1, A2, and -- become from Zn, Mg, nickel, Lu, In, and Sc, B1, and B-2-- are 1 or two or more elements which were chosen from the group which consists of Nb, Ta, Mo, and W. The percentage in a1, a2 --, and a chemical formula for the ionic valency of A1, A2, and --, respectively Y1, Y2 --, B1, B-2 -- The percentage in b1, b2 --, and a chemical formula for ionic valency, respectively Z1, Z2 --, The total W of the ionic valency of the element group in a parenthesis [in / when it carries out / chemical formula Pb (A1Y1a1, A2Y2a2, --, B1Z1b1, and B1Z2b2 --) O3] is W=a1andY1+a2Y2+. -- b1andZ1+b2Z2+ -- Fill the charge of =4+.
(b) Do 0.5 ppm-1 mass % addition of 1 of Mn and Cr**, or two sorts at the above (a).
[Claim 4] The manufacture approach of the piezo-electric single crystal component characterized by performing the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field as polarization conditions for the thickness direction of a piezo-electric single crystal component, and manufacturing a domain control piezo-electricity single crystal component.
[Claim 5] The manufacture approach of the piezo-electric single crystal component according to claim 4 characterized by manufacturing a domain control piezo electric crystal single crystal component by performing the process which impresses electric field in the direction which intersects perpendicularly in the direction of polarization of a single crystal piezoelectric device as a preceding

paragraph story of a process according to claim 4, performing the process which controls the direction of the ferroelectric domain of the direction which intersects perpendicularly in the direction of polarization, and performing the process subsequently to claim 4 indicated.
[Claim 6] The process which carries out heating cooling of the single crystal piezoelectric device as a preceding paragraph story of a process according to claim 4 on both sides of the transition temperature of rhombohedral [which is the hypothermic phase of this piezo-electric single crystal ingredient], and ****** which is a moderate temperature phase (1), Or the process which carries out heating cooling on both sides of the Curie temperature which is ****** of this piezo-electric single crystal ingredient, a ferroelectricity, and piezoelectric disappearance temperature (2), Or the process which carries out heating cooling in the cubic temperature requirement which is a parent phase more than Curie temperature (3), Or by performing the process (4) which combined suitably said process (1), (2), and (3), and performing the process subsequently to claim 4 indicated The manufacture approach of the piezo-electric single crystal component characterized by controlling the direction of the ferroelectric domain of the direction which intersects perpendicularly in the direction of polarization, and manufacturing a domain control piezo electric crystal single crystal component.
[Claim 7] The process which impresses electric field in the direction which intersects perpendicularly in the direction of polarization of a single crystal piezoelectric device, The process which carries out heating cooling of the single crystal piezoelectric device on both sides of the transition temperature of rhombohedral [which is the hypothermic phase of this piezo-electric single crystal ingredient], and ****** which is a moderate temperature phase (1), Or the process which carries out heating cooling on both sides of the Curie temperature which is ****** of this piezo-electric single crystal ingredient, a ferroelectricity, and piezoelectric disappearance temperature (2), Or the process which carries out heating cooling in the temperature requirement of the cube article which is a parent phase more than Curie temperature (3), Or the process which uses together the process (4) which combined suitably said process (1), (2), and (3) is performed. Subsequently, the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field is performed. The manufacture approach of the piezo-electric single crystal component characterized by controlling the direction of the ferroelectric domain of the direction which intersects perpendicularly in the direction of polarization, and manufacturing a domain control piezo electric crystal single crystal component.

[Translation done.]

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1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to a piezo-electric single crystal component and its manufacture approach. In more detail, it is the component which consists of a single crystal ingredient, and is related with the component which paid its attention to the domain control of the direction which intersects perpendicularly in the direction of polarization, i.e., the electromechanical coupling coefficient of the longitudinal direction oscillation mode and this direction, and its manufacture approach.
[0002]
[Description of the Prior Art] About the piezo-electric single crystal component, the ultrasonic probe using the piezo electric crystal which consists of a dissolution single crystal of zinc niobic acid-lead titanate is indicated by JP,6-38963,A, for example. This technique has [such a piezo electric crystal] the electromechanical coupling coefficient (k33) of the direction of polarization as large as 80 - 85%, and by using this single crystal shows that a highly sensitive probe is obtained. Although a piezo-electric single crystal component is studied about the electromechanical coupling coefficient of the direction of polarization in this way and from Seki also of various kinds of applications is carried out conventionally, about the property of the direction which intersects perpendicularly in the direction of polarization, it is an uncivilized technical field.
[0003]
[Problem(s) to be Solved by the Invention] various applications are presented with this invention persons when the electromechanical coupling coefficient (k33) of the direction of polarization of a piezo-electric single crystal component (lengthwise direction oscillation mode) has a >=80% value -- **** -- nevertheless the electromechanical coupling coefficient (k31) of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization -- for example IEEE Proc.MEDICAL As shown in the reference of IMAGING3664(1999) pp.239 or others, as compared with the electromechanical coupling coefficient (k33) of 49% - 62% and the direction of polarization (lengthwise direction oscillation mode), it is a low value. And it noted that the value which has dispersion with reference was shown. And as a result of inquiring wholeheartedly, for 800 or more pC/N and k31, -d31** is [d33] 1200 pC/N (the definition top d31) to coincidence at 70% or more simultaneous [k33] at 80% or more. More than it had a negative value, when it carries out, manufacture of the piezo-electric single crystal component which used k31 effectively is possible. For d33, 800 or more pC/N and k31 are [k33] coincidence at 30% or less to coincidence in 80% or more. - d31 is 300 pC/N (the definition top d31). When it was made the following with a negative value, since there was no spurious (unnecessary vibration) generating into the use band, the value of k33 could be used still more efficiently, and it discovered that the piezo-electric single crystal component of lengthwise direction oscillation mode (k33) use of high performance was obtained more.
[0004] Furthermore, the electromechanical coupling coefficient (k31) of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization while it has a big electromechanical coupling coefficient (k33) in the direction of polarization (lengthwise direction oscillation mode) is small. The domain structure formed of the electric dipole about the direction of polarization of a piezo-electric single crystal component where polarization of the cause

of having dispersion was carried out, and the direction which intersects perpendicularly is not a single domain. It found out that the piezo-electric single crystal component of the following (A) and (B) was obtained that it is because it is formed in two or more domains (multi-domain), and by controlling this domain structure.
[0005] (A) In the piezo-electric single crystal ingredient which is electromechanical coupling coefficient k33 >=80% of the lengthwise direction oscillation mode of the direction of polarization, and has piezo-electric distorted constant d33 >=800 pC/N electromechanical coupling coefficient k31 >=70% of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization -- and It has piezo-electric distorted constant-d31 >=1200 pC/N. And the domain control piezo-electricity single crystal component which is value fc31 <=650 Hz-m of the resonance frequency (fr) of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization about k31, and the frequency constant (fc31=fr-L) which is the product of the oscillating lay length (L) of a component.
[0006] (B) In the piezo-electric single crystal ingredient which is electromechanical coupling coefficient k33 >=80% of the lengthwise direction oscillation mode of the direction of polarization, and has piezo-electric distorted constant d33 >=800 pC/N electromechanical coupling coefficient k31 <=30% of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization -- and It has piezo-electric distorted constant-d31 <=300 pC/N. And the domain control piezo-electricity single crystal component which is value fc31 >=800 Hz-m of the resonance frequency (fr) of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization about k31, and the frequency constant (fc31=fr-L) which is the product of the oscillating lay length (L) of a component.
[0007] Moreover, it discovered that the conditions which control domain structure were arranged with the value of the frequency constant (fc31=fr-L) which is the resonance frequency (fr) of the oscillation mode and the product of the oscillating lay length (L) of a component in connection with the electromechanical coupling coefficient k31 of the direction of polarization of this piezo-electric single crystal component, and the direction (longitudinal direction oscillation mode) which intersects perpendicularly.
[0008] This invention aims at offering such a piezo-electric single crystal component by which domain control was carried out, and its manufacture approach.
[0009]
[Means for Solving the Problem] In the piezo-electric single crystal ingredient which invention of the 1st of this invention is electromechanical coupling coefficient k33 >=80% of the lengthwise direction oscillation mode of the direction of polarization, and has piezo-electric distorted constant d33 >=800 pC/N It is electromechanical coupling coefficient k31 >=70% of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization, and is piezo-electric distorted constant-d31 >=1200 pC/N (the definition top d31). It has. a negative value -- having -- And it is the domain control piezo-electricity single crystal component characterized by being value fc31 <=650 Hz-m of the resonance frequency (fr) of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization about k31, and the frequency constant (fc31=fr-L) which is the product of the oscillating lay length (L) of a component.
[0010] Next, invention of the 2nd of this invention is set into the piezo-electric single crystal ingredient which is electromechanical coupling coefficient k33 >=80% of the lengthwise direction oscillation mode of the direction of polarization, and has piezo-electric distorted constant d33 >=800 pC/N. It is electromechanical coupling coefficient k31 <=30% of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization, and is piezo-electric distorted constant-d31 <=300 pC/N (the definition top d31). It has. a negative value -- having -- And it is the domain control piezo-electricity single crystal component characterized by being value fc31 >=800 Hz-m of the resonance frequency (fr) of the longitudinal direction oscillation mode of the direction which intersects perpendicularly in the direction of polarization about k31, and the frequency constant (fc31=fr-L) which is the product of the oscillating lay length (L) of a component.
[0011] A slenderness ratio makes the longitudinal direction the direction of polarization about three

or more rod-like structures, and the piezo-electric single crystal component expresses vibration (lengthwise direction vibration) of the direction of polarization when applying an electrical potential difference in the direction of polarization, and the conversion efficiency of distorted magnitude with the electromechanical coupling coefficient (k33) and the piezo-electric distorted constant (d33) of the lengthwise direction oscillation mode, respectively, and it is so efficient that these numeric values are large. Its it is specified also about the thing of the configuration of a rectangular plate besides a rod-like structure, a disk, etc. This invention is the domain control piezo electric crystal single crystal component which paid its attention to the electromechanical coupling coefficient (k31) of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization.

[0012] As a piezo-electric single crystal ingredient concerning the 1st above-mentioned invention or the 2nd invention, following (a) or (b) can be used suitably.

[0013] (a) It is the solid solution which consists of X-Pb(A1, A2, --, B1, B-2--) O3+(1-X) PbTiO3 (0< X<1). 1 or two or more elements which were chosen from the group which A1, A2, and -- become from Zn, Mg, nickel, Lu, In, and Sc, B1, and B-2-- are the elements of the 1 and the number of simplicity and complexity which were chosen from the group which consists of Nb, Ta, Mo, and W. The percentage in a1, a2 --, and a chemical formula for the ionic valency of A1, A2, and --, respectively Y1, Y2 --, B1, B-2 -- The percentage in b1, b2 --, and a chemical formula for ionic valency, respectively Z1, Z2 --, The total W of the ionic valency of the element group [in / when it carries out / chemical formula Pb(A1Y1a1, A2Y2a2, --, B1Z1b1, and B1Z2b2 --) O3] in a parenthesis is W=a1andY1+a2Y2+. -- b1andZ1+b2Z2+ -- Fill the charge of =4+.

[0014] (b) Do 0.5 ppm-1 mass % addition of 1 of Mn and Cr**, or two sorts at the above (a).

[0015] In addition, there is a piezo electric crystal single crystal ingredient (PZN-PT or PZNT, and the latter are called PMN-PT or PMNT for the former) which consists of the solid solution of lead-zinc-niobate Pb(Zn1/3Nb 2/3) O3, magnesium lead niobate Pb(Mg1/3Nb 2/3) O3, and lead titanate PbTiO3 as an ingredient known best.

[0016] There is the manufacture approach shown below as an approach of manufacturing the above domain control piezo-electricity single crystal component.

[0017] One of them is the process (electric-field cooling) cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field as polarization conditions for the thickness direction of a piezo-electric single crystal component, and it is the manufacture approach of the piezo-electric single crystal component characterized by manufacturing the above-mentioned domain control piezo-electricity single crystal component.

[0018] Although this manufacture approach is a process which performs final polarization of a domain control single crystal piezoelectric device, its manufacture approach of adding the process which impresses electric field in the direction of polarization and the direction which intersects perpendicularly, and controls the alignment condition of the ferroelectric domain of the direction of polarization and the direction which intersects perpendicularly to the preceding paragraph of this process is also effective. As a class of electric field impressed in the direction of polarization, and the direction which intersects perpendicularly, there are attenuation electric field besides direct-current electric field, pulse electric field, alternating current electric fields, and these stationary electric fields etc., and field strength, impression time amount, temperature conditions, etc. have proper conditions according to the value of a request of the electromechanical coupling coefficient (k31) of the direction which intersects perpendicularly in each property and direction of polarization of a piezo-electric single crystal component. These can be defined by experiment etc. Moreover, as the aforementioned pulse electric field, unipolars, such as an others and alternating current triangular wave, and a bipolar pulse can be used. [wave / right-angle]

[0019] Moreover, the manufacture approach characterized by heating and cooling a single crystal piezoelectric device is in the preceding paragraph of the process which performs final polarization of the domain control single crystal piezoelectric device cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 above-mentioned degrees C temperature requirement for a maximum of 2 hours, or impressing electric field as an option of this invention. For example, the temperature field where a piezo-electric single crystal component serves as

rhombohedral, ******, and a cubic was decided according to the presentation. Therefore, for example, the process which carries out heating cooling of the single crystal piezoelectric device on both sides of the transition temperature of rhombohedral [which is the hypothermic phase of this piezo-electric single crystal ingredient], and ****** which is a moderate temperature phase (1), Or Curie temperature which is ****** of this piezo-electric single crystal ingredient, a ferroelectricity, and piezoelectric disappearance temperature (at an elevated temperature, from this temperature) this ****** single crystal ingredient -- a cubic (parent phase) -- becoming -- the process (2) which inserts and carries out heating cooling -- Or the process which carries out heating cooling within a parent phase (3), Or the process (4) which combines a process (1), (2), and (3) suitably is performed. Subsequently, by performing the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field, the alignment condition of the ferroelectric domain of the direction which intersects perpendicularly in the direction of polarization is controllable.

[0020] In furthermore, the preceding paragraph of the process which performs final polarization of the process domain control single crystal piezoelectric device cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 above-mentioned degrees C temperature requirement for a maximum of 2 hours, or impressing electric field The process which impresses electric field in the direction which intersects perpendicularly in the direction of polarization of a single crystal piezoelectric device, By performing the process which uses together the process which carries out heating cooling of the single crystal piezoelectric device, and performing the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field subsequently in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field The ferroelectric domain alignment condition of the direction which intersects perpendicularly in the direction of polarization is controllable.

[0021]

[Embodiment of the Invention] For example, the solid-solution single crystal of zinc niobic acid-lead titanate (PZN-PT or PZNT) is making the perovskite structure (ABO3) as the unit lattice showed typically to drawing 1 . The phase diagram by the presentation ratio of PZN and PT was shown in drawing 2 . This drawing is Nomura. et at., J.Phys. (1969). J. Kuwata et It quotes from at. and Ferroelectrics (1981). In rhombohedral [PZNT], it has the spontaneous polarization which corresponds in the eight directions of <111> bearings of the crystal when regarding as a pseudo-cubic at an electric dipole so that drawing 2 may see. If electric field are impressed to such a spontaneous polarization condition in the **** <100> direction (the crystal logging direction), an electric dipole will rotate in the polarization electric-field impression direction, and the direction of spontaneous polarization will come to gather.

[0022] However, although various conditions arise in this way of gathering by the mode of impression of electric field etc., consequently the electromechanical coupling coefficient (k33) of the direction of polarization has 80% or more of value in it It turned out that the electromechanical coupling coefficient (k31) of the direction which intersects perpendicularly in the direction of polarization is distributed to 49 - 62% with dispersion according to the reference etc., i.e., control of an electromechanical coupling coefficient (k31) is not made about the direction of polarization, and the direction (transverse-oscillation mode) which intersects perpendicularly. In such a value of k31, it was difficult to produce the device which used k31 positively, or spurious one occurred in the lengthwise direction oscillating (k33) mode of the direction of polarization in the device which uses k33 positively on the other hand, and the situation that sufficient property could not be acquired had occurred. The factor which gives this result is explained as follows. That is, for the material of the piezo-electric single crystal component started from the piezo electric crystal single crystal after training, the domain which consists of a set of the electric dipole of the same direction in the direction of polarization and the direction of polarization, and the direction that intersects perpendicularly has turned to various directions, and does not show piezoelectric, but is in the condition of non-polarization.

[0023] General polarization processing temperature and applied voltage can be chosen, and many domains will not be able to gather without impressing electric field in the direction of polarization in the direction of polarization. By this, the electromechanical coupling coefficient k33 of the direction

of polarization comes to show 80% or more of big value. However, the condition of the domain in the direction of polarization and the direction which intersects perpendicularly can be controlled only within limits with suitable the polarization conditions in the direction of polarization, i.e., polarization processing temperature, and applied voltage.

[0024] Next, an example is given and explained about how to control the mode of polarization. Table 1 shows the dielectricity and the piezo-electric property at the time of changing the polarization conditions of the piezo-electric single crystal ingredient in connection with the conventional example (sample numbers 1, 2, and 3), a reference value (reference values 1 and 2), and this invention etc. d33 value in Table 1 was measured by d33 m (Chinese Academy of Sciences **** lab ZJ-3D mold). It asked for calculation of k33 value from d33vsk33 curve based on this invention persons' measurement shown in drawing 12 . k31, d31, and fc31 measured the frequency response of an impedance, and computed it by count. The piezo-electric single crystal component (component configuration: 13mm die-length x4mm width-of-face x0.36mm thickness) of used 0.91PZN(s) +0.09PT (it expresses with X= 0.91 and a mole fraction) As shown in drawing 3 , the 6th page produces a golden electrode by the spatter to two fields (001) 11 of the crystal 10 surrounded in the field (100) which counter, and it is immersed into a 40-degree C silicone oil. To inter-electrode, 250v [mm] (sample number 4) /and 500v (sample number 5)/mm After impressing each [700v / mm / (sample number 6) /, 1000v / mm / (sample number 7) /, and 1600v //mm / (sample number 8)] electric field for 10 minutes, the impedance curve in the k31 mode was shown in drawing 4 - drawing 8 . It is in the condition that mm is [v / (drawing 4) / // 250] inadequate in polarization, and although three k31 oscillation modes are seen by mm in 500v [mm] (drawing 5) /and 700v (drawing 6) /, this is because there are two or more domains in the direction which intersects perpendicularly in the direction of polarization.

[0025] The domain in the direction which intersects perpendicularly in the direction of polarization is a single domain, and as for the value of k31, k33 of the direction of polarization shows >95% at the same time it fills >80%, so that clearly [v / (drawing 7) //mm / 1000] from an impedance curve. Although it separated into two or more domains again in mm in 1600v (drawing 8) /and the value of k33 was >95%, the value of k31 is 61%. Moreover, the values fc31 of the frequency constant (fc31=fr-L) which is the resonance frequency (fr) of the longitudinal direction oscillation mode and the product of the oscillating lay length (L) of a component about k31 of each sample were [in the sample number 4 / in 741 Hz-m and a sample number 5 / in 601 Hz-m and a sample number 6] 700 Hz-m in 522 Hz-m and a sample number 8 at 603 Hz-m and a sample number 7. The condition of the domain within the field which intersects perpendicularly in the direction of polarization after 250v /, 500v /, 1000v /, and 1600v [/mm] impression mm mm is shown in drawing 9 . [mm]

[0026] In drawing 9 , although mm is [v / // 250] inadequate in polarization and it is two or more domains (multi-domain) in mm in 500v /, k31 becomes large by the synergism of the polarization component in connection with k31. By mm, it becomes a single domain in 1000v /, and by mm, it becomes two or more domains in 1600 morev /, and k31 becomes small by the phase bactericidal action of the polarization component in connection with k31. the inside of this invention -- high -- k33 (d33) -- high -- the domain arrays from which k31 (d31) is obtained were 500v [mm] /and 1000v/mm. When the temperature of a silicone oil was dropped to the room temperature, impressing 400v [/mm] direct-current electric field for the component of the same setup, and a sample number 9 in a 200-degree C silicone oil on the other hand, the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode) was >=80%, and the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization was >70%. fc31 at this time was 609 Hz-m. In the sample number 10, the component of the same setup was immersed into the 60-degree C silicone oil, and 400v [/mm] direct-current electric field were impressed for 120 minutes. Consequently, although the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode) was >95%, the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization was <30%.

[0027] Moreover, although the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode) was >90% in the sample number 11 when 1500v

[/mm] direct-current electric field were impressed to the component of the same setup for 10 minutes, the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization was <30%. fc(s)31 of a sample number 10 and a sample number 11 were 981 Hz-m and 1004 Hz-m, respectively. It is thought that this result comes from the domain array which suppresses longitudinal direction vibration.

[0028] Thus, by setting up polarization conditions (applied voltage, temperature, etc.) appropriately, the domain condition at the time of polarization and the value of k33 and k31 depending on it are controllable. moreover, the thing for which the temperature requirement, the polarization electric-field value range, impression time amount range, and the impression approach of the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field not only in the example shown here but in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field are used -- the above-mentioned example of **** -- ** -- it is checked that same dielectricity and piezo-electric property are acquired.

[0029] Furthermore, it is related with the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field. The process of the depolarization held at 200 degrees C more than Curie temperature for 1 hour is inserted. It is checked also by repeating the process of the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field that the property shown in the 1st invention and the 2nd invention improves. These results The direction which intersects perpendicularly in the direction of polarization as [show / in drawing 10 / when it arranges with the value of the electromechanical coupling coefficient k31 of the (longitudinal direction oscillation mode), the resonance frequency (fr) in the k31 mode, and the frequency constant (fc31=fr-L) that is the product of the oscillating lay length (L) of a component] -- high -- k33 -- high -- the field of k31 -- high -- k33 -- It turned out that a field low [k31] is obtained considering the range of the value of a frequency constant (fc31=fr-L) as an axis of abscissa.

[0030] Although the conventional example and the reference value were also indicated to coincidence at drawing 10 In the conventional example and a reference value, there is a value of the resonance frequency (fr) in the k31 mode and the frequency constant (fc31=fr-L) which is the product of the oscillating lay length (L) of a component in the middle of claims 1 and 2 of this invention. As clarified by this invention, it is in the field where the domain control of the direction of polarization and the direction which intersects perpendicularly is inadequate, therefore it is thought that the value of k31 varies.

[0031] Next, the process which controls the direction of the ferroelectric domain of the direction which impresses electric field in the direction which intersects perpendicularly in the direction of polarization of a single crystal piezoelectric device, and intersects perpendicularly in the direction of polarization, The process which carries out heating cooling of the single crystal piezoelectric device on both sides of the transition temperature of rhombohedral [which is the hypothermic phase of this piezo-electric single crystal ingredient], and ****** which is a moderate temperature phase (1), Or the process which carries out heating cooling on both sides of the Curie temperature which is ****** of this piezo-electric single crystal ingredient, a ferroelectricity, and piezoelectric disappearance temperature (2), Or the process which carries out heating cooling within a parent phase (3), Or the process (4) which combined suitably said process (1), (2), and (3) is performed. Subsequently, the example which performed the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field is **(ed) and explained in Table 2. The measurement of d33 in Table 2, calculation of k33 value, measurement of k31, d31, and fc31, and count are the same as that of Table 1.

[0032] Before the process of the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field in a sample number 12 The golden electrode was produced by the spatter to two fields (010) 13 which intersect perpendicularly with the field (001) 11 of drawing 3 , and counter the component of the same configuration as the above-mentioned single crystal piezo

electric crystal component as shown in drawing 11 , and in the 40-degree C silicone oil, 1000v [/mm] direct-current electric field were impressed for 10 minutes, and were polarized. The process cooled the etching reagent's having removed this golden electrode, producing a golden electrode by the spatter to two fields (001) 11 which is further shown in drawing 3 , and which counter, and impressing 400v/mm - 1500v [/mm] direct-current electric field in the 20 degrees C - 200 degrees C temperature requirement shown in the above-mentioned example for a maximum of 2 hours, or impressing electric field after taking out a component was carried out, and dielectricity and a piezo-electric property were measured.

[0033] Consequently, as shown in the sample number 12 of Table 2, 2810 pC/N was obtained by the piezo-electric distorted constant d33 97.3% with the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode). Moreover, -2380 pC/N was obtained by the piezo-electric distorted constant d31 85.5% with the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization. The value fc31 of the frequency constant (fc31=fr-L) which is the resonance frequency (fr) of the longitudinal direction oscillation mode and the product of the oscillating lay length (L) of a component about k31 was 483 Hz-m.

[0034] Before the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field in sample numbers 13, 14, and 15 The component of the same configuration as the above-mentioned single crystal piezo electric crystal component was immersed into the silicone oil, respectively, and a 3 times temperature rise and descent were further repeated [the temperature requirement (50-90 degrees C and 150-200 degrees C)] for the 200-400-degree C temperature requirement in a cycle of 30 minutes within the temperature tub. Then, the golden electrode was produced by the spatter to two fields (001) 11 which is shown in drawing 3 and which counter, the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in the 20 degrees C - 200 degrees C temperature requirement shown in the above-mentioned example for a maximum of 2 hours, or impressing electric field was carried out, and dielectricity and a piezo-electric property were measured. Consequently, at the sample number 13, 2840 pC/N was obtained by the piezo-electric distorted constant d33 97.5% with the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode).

[0035] Moreover, it was the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization, and 85.3%, by the piezo-electric distorted constant d31, -2360 pC/N was obtained with the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode), and 2880 pC/N was obtained by the piezo-electric distorted constant d33 97.8% at the sample number 14.

[0036] Moreover, it was the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization, and 85.3%, by the piezo-electric distorted constant d31, -2350 pC/N was obtained with the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode), and 2820 pC/N was obtained by the piezo-electric distorted constant d33 97.4% at the sample number 15. Moreover, -2380 pC/N was obtained by the piezo-electric distorted constant d31 85.6%, respectively with the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization. The value fc31 of a frequency constant (fc31=fr-L) was [at 503 Hz-m and a sample number 14] 437 Hz-mm in the sample number 13 in 506 Hz-m and a sample number 15.

[0037] In the sample number 16, 400v [/mm] direct-current electric field were impressed, having produced the golden electrode by the spatter to two fields (010) 13 which intersect perpendicularly with the field (001) 11 of drawing 3 , and counter, as shown in drawing 11 , having been immersed into the silicone oil, and repeating a 3 times temperature rise and descent for a 150-200-degree C temperature requirement in a cycle of 30 minutes. The process cooled the etching reagent's having removed this golden electrode, producing a golden electrode by the spatter to two fields (001) 11 which is further shown in drawing 3 , and which counter, and impressing 400v/mm - 1500v [/mm] direct-current electric field in the 20 degrees C - 200 degrees C temperature requirement shown in

the above-mentioned example for a maximum of 2 hours, or impressing electric field after taking out a component was carried out, and dielectricity and a piezo-electric property were measured. Consequently, 2870 pC/N was obtained by the piezo-electric distorted constant d33 97.8% with the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode). Moreover, -2450 pC/N was obtained by the piezo-electric distorted constant d31 86.0% with the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization. The value fc31 of a frequency constant (fc31=fr-L) was 415 Hz-m.
[0038] In addition, it intersects perpendicularly with the field (001) of drawing 3 , and as another field which counters, after impressing direct-current electric field to face-to-face [of drawing 11] (100), even if it carries out the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field, it is checking that the same effectiveness is acquired.
[0039] Before the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field in a sample number 17 A golden electrode is attached to two fields (010) 13 which intersect perpendicularly with the field (001) 11 of drawing 3 , and counter the component of the same configuration as the above-mentioned single crystal piezo electric crystal component as shown in drawing 11 by the spatter. The peak value of 500v/mm and the bipolar triangular wave electric field of periodic 800msec were impressed for 10 minutes in the 60-degree C silicone oil. The wave of a bipolar triangular wave was shown in drawing 13 . The process cooled the etching reagent's having removed this golden electrode, producing a golden electrode by the spatter to two fields (001) 11 which is further shown in drawing 3 , and which counter, and impressing 400v/mm - 1500v [/mm] direct-current electric field in the 20 degrees C - 200 degrees C temperature requirement shown in the above-mentioned example for a maximum of 2 hours, or impressing electric field after taking out a component was carried out, and dielectricity and a piezo-electric property were measured. Consequently, 2780 pC/N was obtained by the piezo-electric distorted constant d33 97.1% with the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode). Moreover, -230 pC/N was obtained by the piezo-electric distorted constant d31 18.3% with the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization. The value fc31 of a frequency constant (fc31=fr-L) was 863 Hz-m.
[0040] Before the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field in sample numbers 18, 19, and 20 The component of the same configuration as the above-mentioned single crystal piezo electric crystal component was immersed into the silicone oil, respectively, and a 3 times temperature rise and descent were repeated for the temperature requirement (50-90 degrees C, 150-200 degrees C, and 200-400 degrees C) in a cycle of 5 minutes. Then, the golden electrode was produced by the spatter to two fields (001) 11 which is shown in drawing 3 and which counter, the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in the 20 degrees C - 200 degrees C temperature requirement shown in the above-mentioned example for a maximum of 2 hours, or impressing electric field was carried out, and dielectricity and a piezo-electric property were measured.
[0041] consequently -- a sample number 18 -- the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode) -- 97.0% and the piezo-electric distorted constant d33 -- 2760 pC/N -- moreover, -260 pC/N was obtained by the piezo-electric distorted constant d31 18.6% with the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization. a sample number 19 -- the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode) -- 97.3% and the piezo-electric distorted constant d33 -- 2810 pC/N -- moreover, -190 pC/N was obtained by the piezo-electric distorted constant d31 17.8% with the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization.

[0042] a sample number 20 -- the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode) -- 97.2% and the piezo-electric distorted constant d33 -- 2790 pC/N -- moreover, -220 pC/N was obtained by the piezo-electric distorted constant d31 18.2%, respectively with the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization. The value fc31 of a frequency constant (fc31=fr-L) was [at 836 Hz-m and a sample number 19] 847 Hz-m in the sample number 18 in 892 Hz-m and a sample number 20.
[0043] In the sample number 21, 400v [/mm] direct-current electric field were impressed, having produced the golden electrode by the spatter to two fields (010) 13 which intersect perpendicularly with the field (001) 11 of drawing 3 , and counter, as shown in drawing 11 , having been immersed into the silicone oil, and repeating a 3 times temperature rise and descent for a 150-200-degree C temperature requirement in a cycle of 5 minutes. The process cooled the etching reagent's having removed this golden electrode, producing a golden electrode by the spatter to two fields (001) 11 which is further shown in drawing 3 , and which counter, and impressing 400v/mm - 1500v [/mm] direct-current electric field in the 20 degrees C - 200 degrees C temperature requirement shown in the above-mentioned example for a maximum of 2 hours, or impressing electric field after taking out a component was carried out, and dielectricity and a piezo-electric property were measured. Consequently, 2850 pC/N was obtained by the piezo-electric distorted constant d33 97.7% with the electromechanical coupling coefficient k33 of the direction of polarization (lengthwise direction oscillation mode). Moreover, -150 pC/N was obtained by the piezo-electric distorted constant d31 17.6% with the electromechanical coupling coefficient k31 of the direction (longitudinal direction oscillation mode) which intersects perpendicularly in the direction of polarization. The value fc31 of a frequency constant (fc31=fr-L) was 924 Hz-m.
[0044] The process which performs final polarization on the occasion of the above-mentioned domain control piezo-electricity single crystal component, In namely, the preceding paragraph of the process cooled impressing 400v/mm - 1500v [/mm] direct-current electric field in a 20 degrees C - 200 degrees C temperature requirement for a maximum of 2 hours, or impressing electric field How to impress electric field in the direction of polarization, and the direction which intersects perpendicularly, and control the alignment condition of the ferroelectric domain of the direction of polarization, and the direction which intersects perpendicularly, The process which carries out heating cooling on both sides of the transition temperature of rhombohedral [which is the hypothermic phase of a piezo-electric single crystal ingredient], and ****** which is a moderate temperature phase (1), Or Curie temperature which is ****** of a piezo-electric single crystal ingredient, a ferroelectricity, and piezoelectric disappearance temperature (at an elevated temperature, from this temperature) this piezo electric crystal single crystal ingredient -- a cubic (parent phase) -- becoming -- the process (2) which inserts and carries out heating cooling -- Or the process which carries out heating cooling within a parent phase (3), Or the approach of controlling the alignment condition of the ferroelectric domain of the direction which intersects perpendicularly with the direction of polarization according to the process (4) which combined a process (1), (2), and (3) suitably and the process which impresses electric field in the direction which intersects perpendicularly in the direction of polarization of a single crystal piezoelectric device, Enforcing the approach of controlling the alignment condition of the ferroelectric domain of the direction which intersects perpendicularly with the direction of polarization according to the process which uses together the process which carries out heating cooling of the single crystal piezoelectric device Two or more domains under crystal generated in the annealing process at the time of crystal training by carrying out each above-mentioned process The domain structure of the direction which intersects perpendicularly in the direction of polarization in the process which performs final polarization of the domain control single crystal piezoelectric device which should be controlled more nearly human and manufactures the above-mentioned domain control piezo-electricity single crystal component In order to raise the dielectricity and the piezo-electric property as used in the field of effective in order to control more easily and invention of the 1st of this invention, and the 2nd invention, it turned out that it is effective.
[0045]
[Effect of the Invention] The domain control piezo electric crystal single crystal component and its

manufacture approach of this invention Since it is constituted as mentioned above, for d33, 800 or more pC/N and **** k31 are [k33] coincidence at 70% or more to coincidence in 80% or more. - d31 is 1200 pC/N (the definition top d31). More than it had a negative value, when it carries out, manufacture of the piezo-electric single crystal component which used k31 effectively is possible. For d33, 800 or more pC/N and k31 are [k33] coincidence at 30% or less to coincidence in 80% or more. - d31 is 300 pC/N (the definition top d31). When it was made the following with a negative value, since there was no spurious generating into the use band of the oscillation mode of k33, the k33 mode could be used still more efficiently, and it became possible to obtain the piezo-electric single crystal component of lengthwise direction (k33) oscillation mode use of high performance more.

[0046]

[Table 1]

試料番号	分極条件			誘電・圧電特性					適用される請求項等
	温度 ℃	電界E V/mm	時間 min	k_{33} %	d_{33} 10^{-12} C/N	k_{31} %	d_{31} 10^{-12} C/N	fc_{31} Hz・m	
1	20	1800	10	95.6	2550	61.5	−970	701	従来例
2	60	400	150	95.3	2500	48.7	−694	773	従来例
3	100	300	120	94.0	2360	35.0	−520	755	従来例
4	40	250	10	56.0	165	18.9	−224	741	分極不十分
5	40	500	10	84.0	1190	76.0	−1310	601	請求項1
6	40	700	10	87.0	1420	77.1	−1324	603	請求項1
7	40	1000	10	95.3	2500	80.8	−1701	522	請求項1
8	40	1600	10	95.3	2500	60.9	−939	700	請求項1
9	200	400	電界冷却	80.2	960	74.7	−1263	609	請求項1
10	60	400	120	96.9	2740	26.3	−288	981	請求項2
11	20	1500	10	94.6	2300	27.1	−291	1004	請求項2
文献値1				94	2300	53	−1100	-	
文献値2				90	1734	49	−962	680-733 (平均値：707)	

[0047]

[Table 2]

試料番号	処理内容	条件	誘電・圧電特性					対応する請求項
			k_{33} %	d_{33} 10^{-12} C/N	k_{31} %	d_{31} 10^{-12} C/N	fc_{31} Hz・m	
12	請求項5	直流電界 1000V/mm、40℃、10分	97.3	2810	85.5	−2380	483	請求項1
13	請求項6	50〜90℃、30分周期、繰り返し3回	97.5	2840	85.3	−2360	503	請求項1
14	請求項6	150〜200℃、30分周期、繰り返し3回	97.8	2880	85.3	−2350	506	請求項1
15	請求項6	200〜400℃、30分周期、繰り返し3回	97.4	2820	85.6	−2380	437	請求項1
16	請求項7	直流電界 400V/mm、150〜200℃、30分周期、繰り返し3回	97.8	2870	86.0	−2450	415	請求項1
17	請求項5	バイポーラ三角波 500V/mm、周期 800ms、10分	97.1	2780	18.3	−230	863	請求項2
18	請求項6	50〜90℃、5分周期、繰り返し3回	97.0	2760	18.6	−260	836	請求項2
19	請求項6	150〜200℃、5分周期、繰り返し3回	97.3	2810	17.8	−190	892	請求項2
20	請求項6	200〜400℃、5分周期、繰り返し3回	97.2	2790	18.2	−220	847	請求項2
21	請求項7	直流電界 400V/mm、150〜200℃、5分周期、繰り返し3回	97.7	2850	17.6	−150	924	請求項2

[Translation done.]

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the typical perspective view of the crystal structure.
[Drawing 2] It is the phase diagram of PZN-PT (PZNT). .
[Drawing 3] It is the explanatory view of electric-field impression.
[Drawing 4] It is drawing of the impedance curve of k31 oscillation mode.
[Drawing 5] It is drawing of the impedance curve of k31 oscillation mode.
[Drawing 6] It is drawing of the impedance curve of k31 oscillation mode.
[Drawing 7] It is drawing of the impedance curve of k31 oscillation mode.
[Drawing 8] It is drawing of the impedance curve of k31 oscillation mode.
[Drawing 9] It is drawing of the condition of the domain within the direction (thickness direction) side of polarization after electric-field impression.
[Drawing 10] It is the graph of the value of an electromechanical coupling coefficient k31, the resonance frequency (fr) of k31 oscillation mode, and the frequency constant (fc31=fr-L) that is the product of the oscillating lay length (L) of a component.
[Drawing 11] It is the explanatory view of electric-field impression.
[Drawing 12] It is the graph of k33 pair d33.
[Drawing 13] It is the wave form chart of a bipolar triangular wave pulse.
[Description of Notations]
10 Piezo-electric Single Crystal Component
11 Electrode Surface (001)
12 Electric Field
13 Electrode Surface (010)

[Translation done.]

* NOTICES *

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[Translation done.]

* NOTICES *

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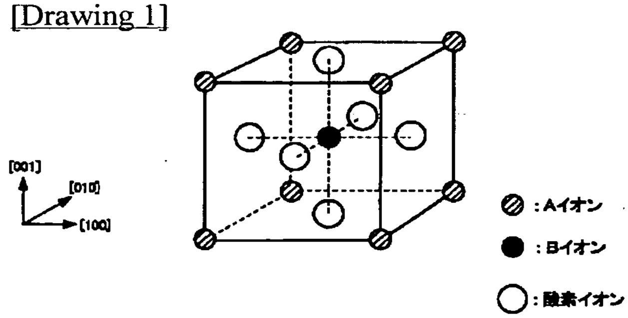
damages caused by the use of this translation.

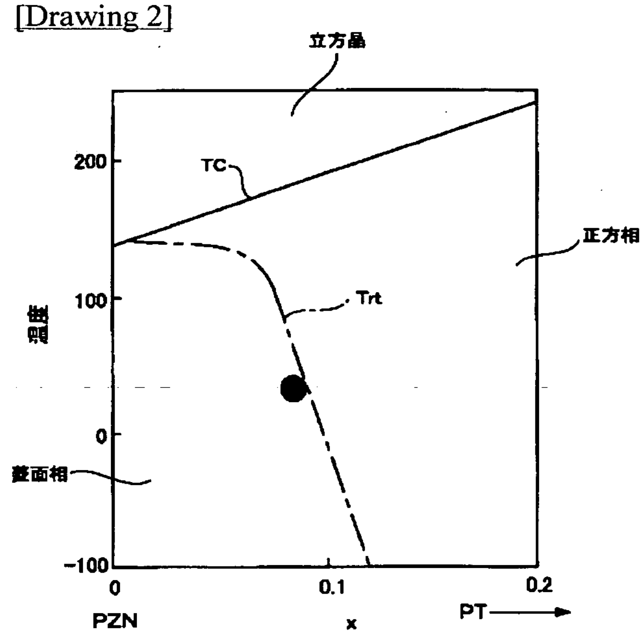
1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DRAWINGS

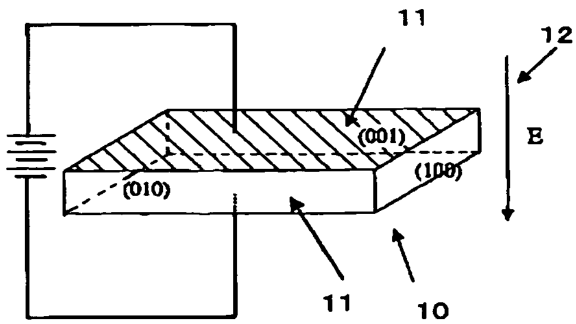
[Drawing 1]



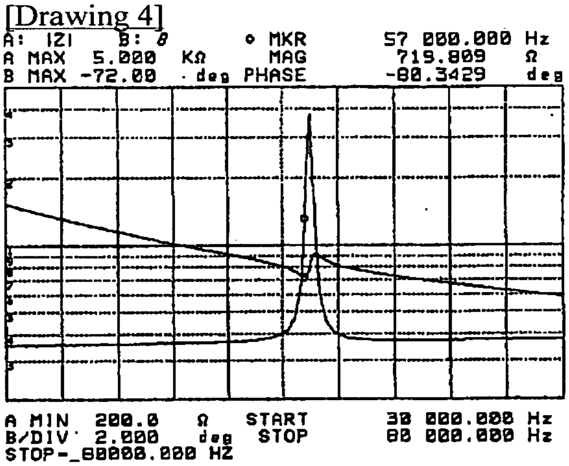
[Drawing 2]



[Drawing 3]

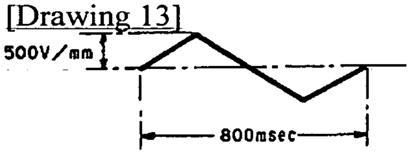


[Drawing 4]

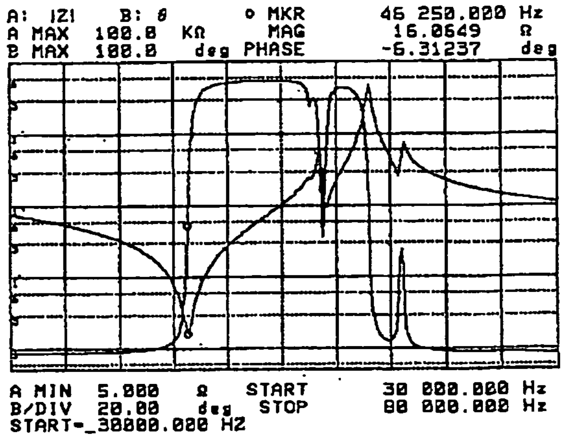


250V/mm

[Drawing 13]

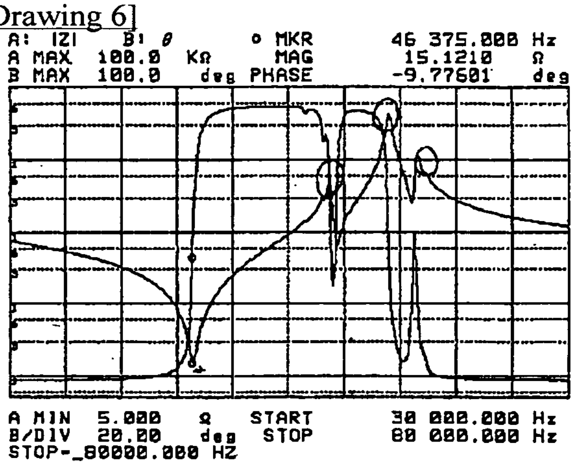


[Drawing 5]



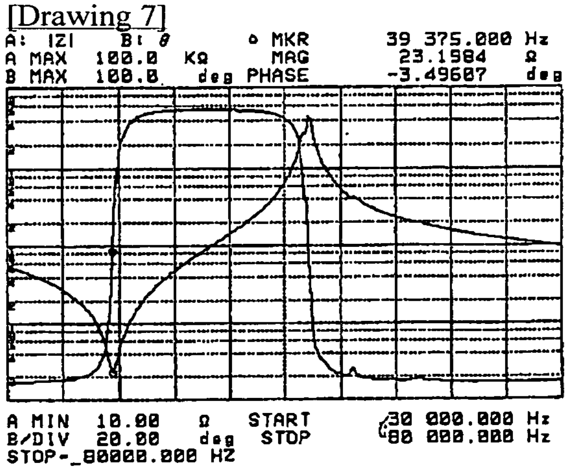
500V/mm

[Drawing 6]



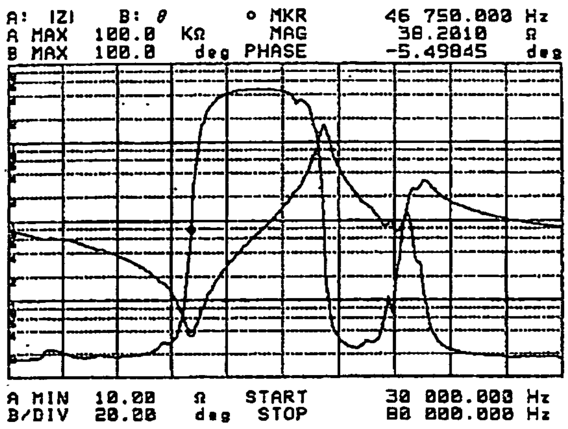
700V/mm

[Drawing 7]



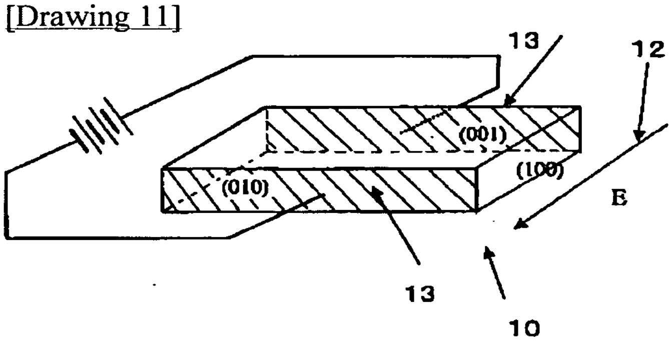
1000V/mm

[Drawing 8]

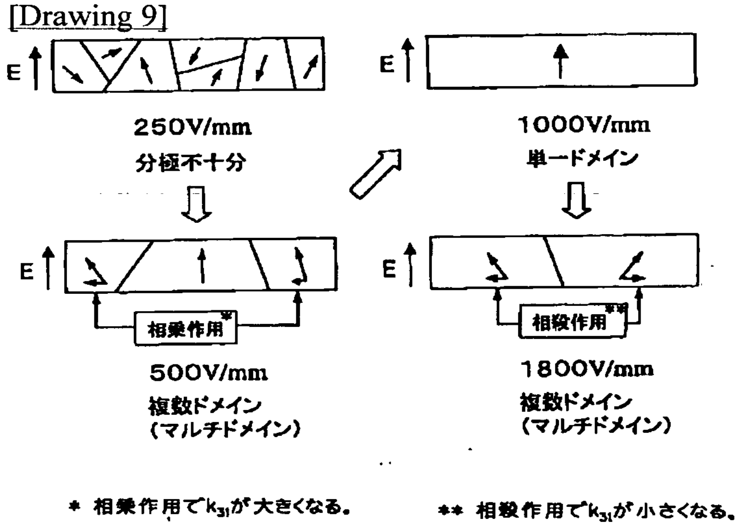


1600 V/mm

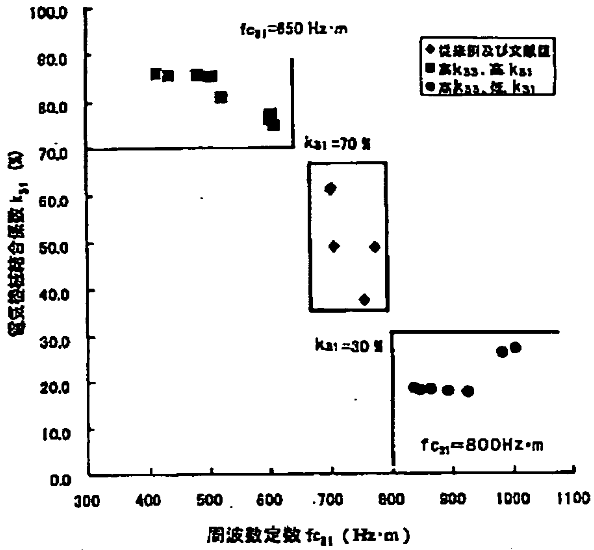
[Drawing 11]



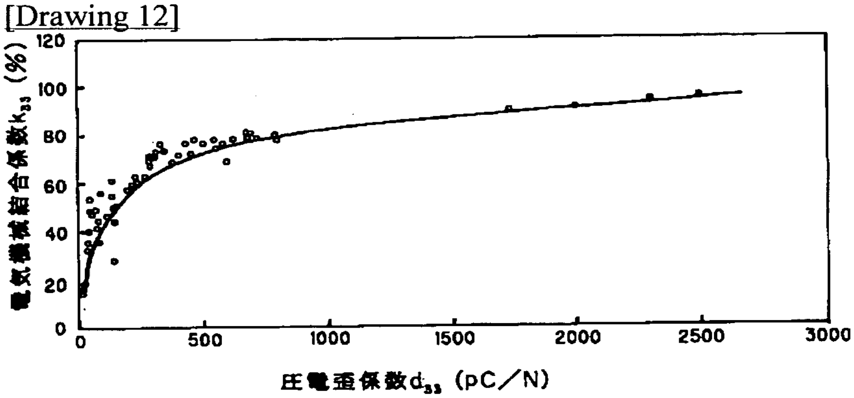
[Drawing 9]



[Drawing 10]



[Drawing 12]



[Translation done.]

(19)日本国特許庁（ＪＰ）　(12) 公開特許公報（Ａ）　(11)特許出願公開番号
特開2003－282986
（P2003－282986A）
(43)公開日　平成15年10月3日(2003.10.3)

(51)Int.Cl.⁷	識別記号	ＦＩ			テーマコード゛(参考)
Ｈ０１Ｌ　41/09		Ｃ０４Ｂ	35/46		Ｍ　４Ｇ０３１
Ｃ０４Ｂ　35/46		Ｃ３０Ｂ	29/32		Ｄ　４Ｇ０７７
Ｃ３０Ｂ　29/32		Ｈ０１Ｌ	41/08		Ｃ
Ｈ０１Ｌ　41/187			41/18	１０１	Ｃ
41/22			41/22		Ｚ

審査請求　未請求　請求項の数７　ＯＬ　（全 11 頁）

(21)出願番号　特願2002－83702(P2002－83702)

(22)出願日　平成14年3月25日(2002.3.25)

特許法第30条第１項適用申請有り　平成13年10月25日～26日　日本セラミックス協会電子材料部会主催の「第２回アジアエレクトロセラミックス会議　第21回電子材料研究討論会」において文書をもって発表

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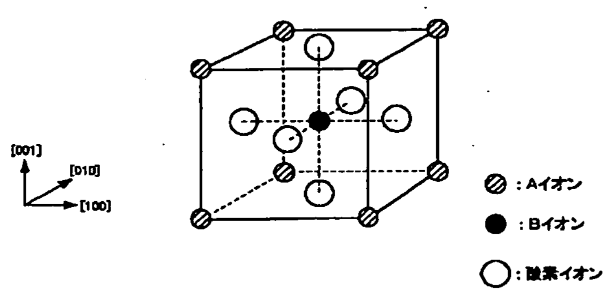
(54)【発明の名称】　ドメイン制御圧電単結晶素子及びその製造方法

(57)【要約】
【課題】k_{33}が８０％以上でd_{33}が８００ｐＣ／Ｎ以上、且つk_{31}が７０％以上で$-d_{31}$が１２００ｐＣ／Ｎ以上のk_{31}を有効に利用した横方向振動モード（k_{31}）利用のドメイン制御圧電単結晶素子、及びk_{33}が８０％以上でd_{33}が８００ｐＣ／Ｎ以上、且つk_{31}が３０％以下でk_{33}振動モードの使用帯域内にスプリアス等のない、高効率、高性能の縦方向振動モード（k_{33}）利用のドメイン制御圧電単結晶素子を開発した。
【解決手段】圧電単結晶素子の厚み方向の分極条件として、２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか、電界を印加したまま冷却すること（電界冷却）、この工程の前段として分極方向に直交する方向に電界を印加すること（電界印加）、又は圧電単結晶の菱面晶－正方晶相境界温度、又は正方晶－立方晶相境界温度を挟んで昇降温するか、又は立方晶温度域の異なる２温度間で昇降温する（熱処理）こと、又は、これらを併用する。



【特許請求の範囲】

【請求項１】　分極方向の縦方向振動モードの電気機械結合係数 k_{33}≧８０％で且つ圧電歪定数 d_{33}≧８００ｐＣ／Ｎを持つ圧電単結晶材料において、分極方向に直交する方向の横方向振動モードの電気機械結合係数 k_{31}≧７０％で且つ、圧電歪定数－d_{31}≧１２００ｐＣ／Ｎを持ち、且つ k_{31} に関する分極方向に直交する方向の横方向振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（fc_{31}＝ｆｒ・Ｌ）の値 fc_{31}≦６５０Ｈｚ・ｍであることを特徴とするドメイン制御圧電単結晶素子。

【請求項２】　分極方向の縦方向振動モードの電気機械結合係数 k_{33}≧８０％で且つ圧電歪定数 d_{33}≧８００ｐＣ／Ｎを持つ圧電単結晶材料において、分極方向に直交する方向の横方向振動モードの電気機械結合係数 k_{31}≦３０％で且つ、圧電歪定数－d_{31}≦３００ｐＣ／Ｎを持ち、且つ k_{31} に関する分極方向に直交する方向の横方向振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（fc_{31}＝ｆｒ・Ｌ）の値 fc_{31}≧８００Ｈｚ・ｍであることを特徴とするドメイン制御圧電単結晶素子。

【請求項３】　圧電単結晶材料が、下記（ａ）又は（ｂ）であることを特徴とする請求項１又は請求項２記載のドメイン制御圧電体単結晶素子。

（ａ）$X \cdot Pb(A_1, A_2, \cdots, B_1, B_2 \cdots)O_3 + (1-X)PbTiO_3$（$0<X<1$）からなる固溶体であって、$A_1$, A_2, …はＺｎ，Ｍｇ，Ｎｉ，Ｌｕ，Ｉｎ及びＳｃからなる群から選ばれた１又は複数の元素、B_1, B_2…はＮｂ，Ｔａ，Ｍｏ及びＷからなる群から選ばれた１又は複数の元素で、A_1, A_2, …のイオン価をそれぞれ a_1, a_2…、化学式中の構成比を Y_1, Y_2…、B_1, B_2…のイオン価をそれぞれ b_1, b_2…，化学式中の構成比を Z_1, Z_2…、とした時に、化学式 $Pb(A_{1Y1}^{a1}, A_{2Y2}^{a2}, \cdots, B_{1Z1}^{b1}, B_{1Z2}^{b2} \cdots)O_3$ におけるかっこ内の元素群のイオン価の総和ＷがＷ＝$a_1 \cdot Y_1 + a_2Y_2 + \cdots b_1 \cdot Z_1 + b_2Z_2 + \cdots = 4+$ の電荷を満たすものであること。

（ｂ）上記（ａ）に、Ｍｎ，Ｃｒ、の１又は２種を０．５ｐｐｍ～１質量％添加したものであること。

【請求項４】　圧電単結晶素子の厚み方向の分極条件として、２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を行い、ドメイン制御圧電単結晶素子を製造することを特徴とする圧電単結晶素子の製造方法。

【請求項５】　請求項４に記載の工程の前段階として、単結晶圧電素子の分極方向に直交する方向に電界を印加する工程を行い、分極方向に直交する方向の強誘電体ドメインの方向を制御する工程を行い、次いで請求項４に記載する工程を行うことによりドメイン制御圧電体単結晶素子を製造することを特徴とする請求項４記載の圧電単結晶素子の製造方法。

【請求項６】　請求項４に記載の工程の前段階として、単結晶圧電素子を該圧電単結晶材料の低温相である菱面晶と中温相である正方晶の転移温度を挟んで加熱冷却する工程（１）、又は該圧電単結晶材料の正方晶と強誘電性・圧電性の消失温度であるキュリー温度を挟んで加熱冷却する工程（２）、又は、キュリー温度以上の高温相である立方晶の温度範囲内で加熱冷却する工程（３）、又は、前記工程（１）、（２）、（３）を適宜組み合わせた工程（４）を行い、次いで請求項４に記載する工程を行うことにより、分極方向に直交する方向の強誘電体ドメインの方向を制御し、ドメイン制御圧電体単結晶素子を製造することを特徴とする圧電単結晶素子の製造方法。

【請求項７】　単結晶圧電素子の分極方向に直交する方向に電界を印加する工程と、単結晶圧電素子を該圧電単結晶材料の低温相である菱面晶と中温相である正方晶の転移温度を挟んで加熱冷却する工程（１）、又は該圧電単結晶材料の正方晶と強誘電性・圧電性の消失温度であるキュリー温度を挟んで加熱冷却する工程（２）、又は、キュリー温度以上の高温相である立方晶の温度範囲内で加熱冷却する工程（３）、又は、前記工程（１）、（２）、（３）を適宜組み合わせた工程（４）を併用する工程を行い、次いで２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を行い、分極方向に直交する方向の強誘電体ドメインの方向を制御し、ドメイン制御圧電体単結晶素子を製造することを特徴とする圧電単結晶素子の製造方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、圧電単結晶素子及びその製造方法に関する。さらに詳しくは、単結晶材料からなる素子であって、分極方向に直交する方向、即ち横方向振動モードの電気機械結合係数と該方向のドメイン制御に着目した素子及びその製造方法に関する。

【０００２】

【従来の技術】圧電単結晶素子については、例えば、特開平６－３８９６３号公報には、亜鉛ニオブ酸－チタン酸鉛の固溶単結晶からなる圧電体を用いた超音波プローブが開示されている。この技術は、このような圧電体が分極方向の電気機械結合係数（k_{33}）が８０～８５％と大きく、この単結晶を使用することにより、感度の良いプローブが得られることを示している。従来、圧電単結晶素子はこのように分極方向の電気機械結合係数について研究され、各種の用途も開発されているが、分極方向に直交する方向の特性については、未開拓の技術分野である。

【０００３】

【発明が解決しようとする課題】本発明者らは、圧電単結晶素子の分極方向（縦方向振動モード）の電気機械結合係数（k_{33}）が≧８０％の値を持つことにより、多種の用途に供されているにもにも拘わらず、分極方向に直交する方向（横方向振動モード）の電気機械結合係数（k_{31}）が例えば、ＩＥＥＥ Ｐｒｏｃ．ＭＥＤＩＣＡＬ ＩＭＡＧＩＮＧ３６６４（１９９９）ｐｐ．２３９やその他の文献に示されるように４９％～６２％と分極方向（縦方向振動モード）の電気機械結合係数（k_{33}）に比較して低い値であり、且つ文献によりばらつきのある値を示すことに着目した。そして鋭意研究した結果、k_{33}が８０％以上で同時にd_{33}が８００ｐＣ／Ｎ以上、且つk_{31}が７０％以上で同時に$-d_{31}$、が１２００ｐＣ／Ｎ（定義上d_{31}は、負の値を持つ）以上にした場合は、k_{31}を有効に利用した圧電単結晶素子の製造が可能であり、k_{33}が８０％以上で同時にd_{33}が８００ｐＣ／Ｎ以上、且つk_{31}が３０％以下で同時に$-d_{31}$が３００ｐＣ／Ｎ（定義上d_{31}は、負の値を持つ）以下にした場合には、k_{33}の値をその使用帯域内にスプリアス（不要振動）等の発生がないため更に効率よく利用でき、より高性能の縦方向振動モード（k_{33}）利用の圧電単結晶素子が得られることを発見した。

【０００４】更に、分極方向（縦方向振動モード）で大きな電気機械結合係数（k_{33}）を有しながら分極方向に直交する方向（横方向振動モード）の電気機械結合係数（k_{31}）が小さく、ばらつきを有する原因は、分極された圧電単結晶素子の分極方向と直交する方向に関する電気双極子により形成されるドメイン構造が単一ドメインでなく、複数個のドメイン（マルチドメイン）で形成されているためであること、そして、該ドメイン構造を制御することにより、次の（Ａ）、（Ｂ）の圧電単結晶素子が得られることを見出した。

【０００５】（Ａ）分極方向の縦方向振動モードの電気機械結合係数k_{33}≧８０％で且つ圧電歪定数d_{33}≧８００ｐＣ／Ｎを持つ圧電単結晶材料において、分極方向に直交する方向の横方向振動モードの電気機械結合係数k_{31}≧７０％で且つ、圧電歪定数$-d_{31}$≧１２００ｐＣ／Ｎを持ち、且つk_{31}に関する分極方向に直交する方向の横方向振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（fc_{31}＝ｆｒ・Ｌ）の値fc_{31}≦６５０Ｈｚ・ｍであるドメイン制御圧電単結晶素子。

【０００６】（Ｂ）分極方向の縦方向振動モードの電気機械結合係数k_{33}≧８０％で且つ圧電歪定数d_{33}≧８００ｐＣ／Ｎを持つ圧電単結晶材料において、分極方向に直交する方向の横方向振動モードの電気機械結合係数k_{31}≦３０％で且つ、圧電歪定数$-d_{31}$≦３００ｐＣ／Ｎを持ち、且つk_{31}に関する分極方向に直交する方向の横方向振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（fc_{31}＝ｆｒ・Ｌ）の値fc_{31}≧８００Ｈｚ・ｍであるドメイン制御圧電単結晶素子。

【０００７】また、ドメイン構造を制御する条件が該圧電単結晶素子の分極方向と直交する方向（横方向振動モード）の電気機械結合係数k_{31}に関わる振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（fc_{31}＝ｆｒ・Ｌ）の値により整理されることを発見した。

【０００８】本発明は、このようなドメイン制御された圧電単結晶素子及びその製造方法を提供することを目的とする。

【０００９】

【課題を解決するための手段】本発明の第１の発明は、分極方向の縦方向振動モードの電気機械結合係数k_{33}≧８０％で且つ圧電歪定数d_{33}≧８００ｐＣ／Ｎを持つ圧電単結晶材料において、分極方向に直交する方向の横方向振動モードの電気機械結合係数k_{31}≧７０％で且つ、圧電歪定数$-d_{31}$≧１２００ｐＣ／Ｎ（定義上d_{31}は、負の値を持つ）を持ち、且つk_{31}に関する分極方向に直交する方向の横方向振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（fc_{31}＝ｆｒ・Ｌ）の値fc_{31}≦６５０Ｈｚ・ｍであることを特徴とするドメイン制御圧電単結晶素子である。

【００１０】次に、本発明の第２の発明は、分極方向の縦方向振動モードの電気機械結合係数k_{33}≧８０％で且つ圧電歪定数d_{33}≧８００ｐＣ／Ｎを持つ圧電単結晶材料において、分極方向に直交する方向の横方向振動モードの電気機械結合係数k_{31}≦３０％で且つ、圧電歪定数$-d_{31}$≦３００ｐＣ／Ｎ（定義上d_{31}は、負の値を持つ）を持ち、且つk_{31}に関する分極方向に直交する方向の横方向振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（fc_{31}＝ｆｒ・Ｌ）の値fc_{31}≧８００Ｈｚ・ｍであることを特徴とするドメイン制御圧電単結晶素子である。

【００１１】圧電単結晶素子は、例えば、細長比が３以上の棒状体について、その長手方向を分極方向とし、分極方向に電圧をかけた時の分極方向の振動（縦方向振動）及び歪の大きさの変換効率をそれぞれ縦方向振動モードの電気機械結合係数（k_{33}）及び圧電歪定数（d_{33}）で表わしており、これらの数値が大きいほど効率が良い。棒状体のほか、方形板や円板等の形状のものについてもそれそれが規定されている。本発明は分極方向に直交する方向（横方向振動モード）の電気機械結合係数（k_{31}）に着目したドメイン制御圧電体単結晶素子である。

【００１２】上記第１の発明又は第２の発明に係る圧電単結晶材料としては、下記（ａ）又は（ｂ）を好適に用いることができる。

【００１３】（ａ）$X \cdot Pb(A_1, A_2, \cdots, B_1, B_2 \cdots)O_3 + (1-X)PbTiO_3$（０＜Ｘ＜１）からな

る固溶体であって、A_1，A_2，…はＺｎ，Ｍｇ，Ｎｉ，Ｌｕ，Ｉｎ及びＳｃからなる群から選ばれた１又は複数の元素、B_1，B_2…はＮｂ，Ｔａ，Ｍｏ及びＷからなる群から選ばれた１又単複数の元素で、A_1，A_2，…のイオン価をそれぞれa_1，a_2…、化学式中の構成比をY_1，Y_2…、B_1，B_2…のイオン価をそれぞれb_1，b_2…，化学式中の構成比をZ_1，Z_2…、とした時に、化学式$Pb(A_{1Y1}{}^{a1}, A_{2Y2}{}^{a2}, \cdots, B^{1Z1b1}, B_{1Z2}{}^{b2\cdots})O_3$における、かっこ内の元素群のイオン価の総和Ｗが$W = a_1 \cdot Y_1 + a_2 Y_2 + \cdots b_1 \cdot Z_1 + b_2 Z_2 + \cdots = 4+$の電荷を満たすものであること。

【００１４】（ｂ）上記（ａ）に、Ｍｎ，Ｃｒ、の１又は２種を０．５ｐｐｍ～１質量％添加したものであること。

【００１５】なお、最もよく知られている材料として亜鉛ニオブ酸鉛$Pb(Zn_{1/3}Nb_{2/3})O_3$やマグネシウムニオブ酸鉛$Pb(Mg_{1/3}Nb_{2/3})O_3$とチタン酸鉛$PbTiO_3$の固溶体からなる圧電体単結晶材料（前者をＰＺＮ－ＰＴ又はＰＺＮＴ、後者をＰＭＮ－ＰＴ又はＰＭＮＴと呼称する）がある。

【００１６】以上のドメイン制御圧電単結晶素子を製造する方法として次に示す製造方法がある。

【００１７】その一つは、圧電単結晶素子の厚み方向の分極条件として、２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか、又は電界を印加したまま冷却する（電界冷却）工程で、上記ドメイン制御圧電単結晶素子を製造することを特徴とする圧電単結晶素子の製造方法である。

【００１８】この製造方法は、ドメイン制御単結晶圧電素子の最終的な分極を行う工程であるが、この工程の前段に、分極方向と直交する方向に電界を印加し、分極方向と直交する方向の強誘電体ドメインの整列状態を制御する工程を加える製造方法も有効である。分極方向と直交する方向に印加する電界の種類としては、直流電界、パルス電界、交流電界、またこれらの定常電界のほか、減衰電界などがあり、電界の強さや印加時間、温度条件等は個々の圧電単結晶素子の特性及び分極方向に直交する方向の電気機械結合係数（k_{31}）の所望の値に応じて適正条件がある。これらは、実験等によって定めることができる。また、前記のパルス電界としては、直角波のほか、交流三角波などユニポーラ及びバイポーラパルスを用いることができる。

【００１９】また、本発明の別の方法として、上記２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか、又は電界を印加したまま冷却するドメイン制御単結晶圧電素子の最終的な分極を行う工程の前段に、単結晶圧電素子を加熱・冷却することを特徴とする製造方法がある。例えば、圧電単結晶素子は、菱面晶、正方晶、立方晶となる温度領域が組成に応じて決まっている。従って、例えば単結晶圧電素子を該圧電単結晶材料の低温相である菱面晶と中温相である正方晶の転移温度を挟んで加熱冷却する工程（１）、又は該圧電単結晶材料の正方晶と強誘電性・圧電性の消失温度であるキュリー温度（この温度より高温では、該年電体単結晶材料は立方晶（高温相）となる）を挟んで加熱冷却する工程（２）、又は、高温相内で加熱冷却する工程（３）、又は、工程（１）、（２）、（３）を適宜組み合わせる工程（４）を行い、次いで２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を行うことにより、分極方向に直交する方向の強誘電体ドメインの整列状態を制御することができる。

【００２０】更に、上記２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程ドメイン制御単結晶圧電素子の最終的な分極を行う工程の前段に、単結晶圧電素子の分極方向に直交する方向に電界を印加する工程と、単結晶圧電素子を加熱冷却する工程とを併用する工程を行い、次いで２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を行うことにより、分極方向に直交する方向の強誘電体ドメイン整列状態を制御することができる。

【００２１】

【発明の実施の形態】例えば、亜鉛ニオブ酸－チタン酸鉛（ＰＺＮ－ＰＴまたはＰＺＮＴ）の固溶体単結晶は、その単位格子が図１に模式的に示したようなペロブスカイト構造（ABO_3）をなしている。図2にＰＺＮとＰＴの組成比による相図を示した。この図はＮｏｍｕｒａ ｅｔ ａｔ．，Ｊ．Ｐｈｙｓ．（１９６９）。Ｊ．Ｋｕｗａｔａ ｅｔ ａｔ．，Ｆｅｒｒｏｅｌｅｃｔｒｉｃｓ（１９８１）より引用したものである。図2に見られるように、菱面晶ＰＺＮＴでは、擬立方晶と見た時の結晶の〈１１１〉方位の８つの方向に電気双極子に相当する自発分極を有している。このような自発分極状態におげる〈１００〉方向（結晶切り出し方向）に電界を印加すると、電気双極子は分極電界印加方向に回転し、自発分極方向が揃うようになる。

【００２２】しかし、この揃い方には、電界の印加の態様等により種々の状態が生じ、その結果、分極方向の電気機械結合係数（k_{33}）が８０％以上の値を持つにもかかわらず、分極方向に直交する方向の電気機械結合係数（k_{31}）が、文献等によれば４９～６２％にばらつきを持って分布すること、即ち、分極方向と直交する方向（横振動モード）に関して電気機械結合係数（k_{31}）の制御がなされていないことがわかった。このようなk_{31}の値では、積極的にk_{31}を利用したデバイスを作製することが困難であったり、一方、積極的にk_{33}を利用するデバイスでは分極方向の縦方向振動（k_{33}）モード中に

スプリアスが発生したりして、十分な特性を得られない状況が発生していた。この結果を与える要因は、以下のように説明される。即ち、育成後の圧電体単結晶から切り出された圧電単結晶素子の素材では、分極方向及び分極方向と直交する方向において同一方向の電気双極子の集合からなるドメインが種々の方向を向いており、圧電性を示さず、未分極の状態にある。

【００２３】一般的な分極処理温度と印加電圧を選択し、分極方向に電界を印加することにより初めて、多くのドメインが分極方向に揃っていくことができる。このことにより、分極方向の電気機械結合係数k_{33}は８０％以上の大きな値を示すようになる。しかし、分極方向と直交する方向におけるドメインの状態は、分極方向での分極条件、即ち、分極処理温度と印加電圧の適切な範囲内でのみ制御することが可能である。

【００２４】次に、分極の態様を制御する方法について、実施例をあげて説明する。表１は従来例（試料番号１，２，３）及び文献値（文献値１，２）及び本発明に関わる圧電単結晶材料の分極条件等を変えた場合の誘電・圧電特性を示したものである。表１中のd_{33}値はd_{33}メータ（中国科学院声学研究所製ＺＪ－３Ｄ型）で測定した。k_{33}値の算出は、本発明者らの測定に基づく、図１２に示すd_{33}ｖｓk_{33}カーブから求めた。k_{31}、d_{31}、fc_{31}はインピーダンスの周波数応答を測定し、計算により算出した。使用した０．９１ＰＺＮ＋０．０９ＰＴ（Ｘ＝０．９１とモル分率で表現）の圧電単結晶素子（素子形状：１３mm長さ×４mm幅×０．３６mm厚み）を、図３に示すように６面が（１００）面で囲まれた結晶１０の二つの対向する（００１）面１１に金電極をスパッタ法で作製し、４０℃のシリコンオイル中に浸漬して、電極間に２５０Ｖ／mm（試料番号４）、５００Ｖ／mm（試料番号５）、７００Ｖ／mm（試料番号６）、１０００Ｖ／mm（試料番号７）、１６００Ｖ／mm（試料番号８）の各電界を１０分間印加した後での、k_{31}モードのインピーダンスカーブを図４～図８に示した。２５０Ｖ／mm（図４）では、分極不十分の状態であり、５００Ｖ／mm（図５）、７００Ｖ／mm（図６）では、３つのk_{31}振動モードが見られるが、これは分極方向に直交する方向に複数のドメインがあるためである。

【００２５】１０００Ｖ／mm（図７）では、インピーダンスカーブから明らかなように、分極方向に直交する方向でのドメインは単一ドメインとなっており、k_{31}の値は＞８０％を満たすと同時に分極方向のk_{33}も＞９５％を示す。１６００Ｖ／mm（図８）では、再び、２つ以上のドメインに分離し、k_{33}の値は＞９５％であったが、k_{31}の値は６１％である。又、それぞれの試料のk_{31}に関する横方向振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（fc_{31}＝ｆｒ・Ｌ）の値fc_{31}は、試料番号４で７４１Ｈｚ・ｍ、試料番号５で６０１Ｈｚ・ｍ、試料番号６で６０３Ｈｚ・ｍ、試料番号７で５２２Ｈｚ・ｍ、試料番号８で７００Ｈｚ・ｍであった。２５０Ｖ／mm、５００Ｖ／mm、１０００Ｖ／mm、１６００Ｖ／mm印加後の分極方向に直交する面内のドメインの状態を図９に示す。

【００２６】図９において、２５０Ｖ／mmでは分極不十分であり、５００Ｖ／mmでは複数ドメイン（マルチドメイン）であるが、k_{31}に関わる分極成分の相乗作用でk_{31}が大きくなる。１０００Ｖ／mmでは単一ドメインとなり、さらに１６００Ｖ／mmでは複数ドメインとなり、k_{31}に関わる分極成分の相殺作用でk_{31}が小さくなる。本発明内で高k_{33}（d_{33}）、高k_{31}（d_{31}）が得られるドメイン配列は５００Ｖ／mm、１０００Ｖ／mmであった。一方、同様な設定の素子、試料番号９を２００℃のシリコンオイル中で、４００Ｖ／mmの直流電界を印加したまま、シリコンオイルの温度を室温まで降下させると、分極方向（縦方向振動モード）の電気機械結合係数k_{33}は≧８０％であり、分極方向に直交する方向（横方向振動モード）の電気機械結合係数k_{31}は＞７０％であった。この時のfc_{31}は６０９Ｈｚ・ｍであった。試料番号１０では、同じ設定の素子を６０℃のシリコンオイル中に浸漬し、４００Ｖ／mmの直流電界を１２０分間印加した。その結果、分極方向（縦方向振動モード）の電気機械結合係数k_{33}は＞９５％であったが、分極方向に直交する方向（横方向振動モード）の電気機械結合係数k_{31}は＜３０％であった。

【００２７】又、試料番号１１では、同じ設定の素子に１５００Ｖ／mmの直流電界を１０分間印加すると、分極方向（縦方向振動モード）の電気機械結合係数k_{33}は＞９０％であったが、分極方向に直交する方向（横方向振動モード）の電気機械結合係数k_{31}は＜３０％であった。試料番号１０及び試料番号１１のfc_{31}は、それぞれ９８１Ｈｚ・ｍ及び１００４Ｈｚ・ｍであった。この結果は、横方向振動を抑えるドメイン配列からくるものと考えられる。

【００２８】このように分極条件（印加電圧、温度等）を適切に設定することにより、分極時のドメイン状態及び、それに依存するk_{33}，k_{31}の値を制御することができる。又、ここに示した実施例に限らず、２０℃～２００℃の温度範囲で４００Ｖ／mm～１５００Ｖ／mmの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程の温度範囲、分極電界値範囲、印加時間範囲と印加方法を用いることにより、上記案施例にと同様な誘電・圧電特性が得られることが確認されている。

【００２９】更に、２０℃～２００℃の温度範囲で４００Ｖ／mm～１５００Ｖ／mmの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程に関しては、キュリー温度以上の２００℃で１時間保持する脱分極の工程を挟んで、２０℃～２００℃の温度範囲で４

００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程の工程を繰り返すことによっても第１の発明、第２の発明に示す特性が向上することが確認されている。これらの結果を、分極方向に直交する方向（横方向振動モード）の電気機械結合係数ｋ$_{31}$とｋ$_{31}$モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（ｆｃ$_{31}$＝ｆｒ・Ｌ）の値で整理すると図１０に示すような高ｋ$_{33}$、高ｋ$_{31}$の領域と高ｋ$_{33}$、低ｋ$_{31}$の領域が周波数定数（ｆｃ$_{31}$＝ｆｒ・Ｌ）の値の範囲を横軸として得られることが解った。

【００３０】従来例及び文献値も同時に図１０に記載したが、従来例及び文献値ではｋ$_{31}$モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（ｆｃ$_{31}$＝ｆｒ・Ｌ）の値が本発明の請求項１、２の中間にあり、本発明で明らかにしたように分極方向と直交する方向のドメイン制御が不充分な領域にあり、そのため、ｋ$_{31}$の値がばらつくものと考えられる。

【００３１】次に、単結晶圧電素子の分極方向に直交する方向に電界を印加し、分極方向に直交する方向の強誘電体ドメインの方向を制御する工程、単結晶圧電素子を該圧電単結晶材料の低温相である菱面晶と中温相である正方晶の転移温度を挟んで加熱冷却する工程（１）、又は該圧電単結晶材料の正方晶と強誘電性・圧電性の消失温度であるキュリー温度を挟んで加熱冷却する工程（２）、又は、高温相内で加熱冷却する工程（３）、又は、前記工程（１）、（２）、（３）を適宜組み合わせた工程（４）を行い、次いで２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を行った実施例について表２に則して説明する。表２中のｄ$_{33}$の測定、ｋ$_{33}$値の算出、ｋ$_{31}$、ｄ$_{31}$、ｆｃ$_{31}$の測定及び計算は表１と同様である。

【００３２】試料番号１２では、２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程の工程の前に、上記の単結晶圧電体素子と同じ形状の素子に、図１１に示すように図３の（００１）面１１と直交して対向する２つの（０１０）面１３に金電極をスパッタ法で作製し、４０℃のシリコンオイル中で１０００Ｖ／ｍｍの直流電界を１０分間印加し分極した。素子を取り出したあと、エッチング液で該金電極を除去し、更に図３に示す二つの対向する（００１）面１１に金電極をスパッタ法で作製し、上記実施例で示した２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を実施し、誘電・圧電特性を測定した。

【００３３】その結果、表２の試料番号１２に示すように、分極方向（縦方向振動モード）の電気機械結合係数ｋ$_{33}$で９７．３％、圧電歪定数ｄ$_{33}$で２８１０ｐＣ／Ｎを得た。又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数ｋ$_{31}$で８５．５％、圧電歪定数ｄ$_{31}$で－２３８０ｐＣ／Ｎを得た。ｋ$_{31}$に関する横方向振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（ｆｃ$_{31}$＝ｆｒ・Ｌ）の値ｆｃ$_{31}$は、４８３Ｈｚ・ｍであった。

【００３４】試料番号１３、１４、１５では、２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程の前に、上記の単結晶圧電体素子と同じ形状の素子を、それぞれシリコンオイル中に浸漬し、５０～９０℃、１５０～２００℃の温度範囲を、更に温度槽内で２００～４００℃の温度範囲を３０分周期で３回温度上昇・下降を繰り返した。その後、図３に示す二つの対向する（００１）面１１に金電極をスパッタ法で作製し、上記実施例で示した２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を実施し、誘電・圧電特性を測定した。その結果、試料番号１３では、分極方向（縦方向振動モード）の電気機械結合係数ｋ$_{33}$で９７．５％、圧電歪定数ｄ$_{33}$で２８４０ｐＣ／Ｎを得た。

【００３５】又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数ｋ$_{31}$で８５．３％、圧電歪定数ｄ$_{31}$で－２３６０ｐＣ／Ｎを、試料番号１４では分極方向（縦方向振動モード）の電気機械結合係数ｋ$_{33}$で９７．８％、圧電歪定数ｄ$_{33}$で２８８０ｐＣ／Ｎを得た。

【００３６】又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数ｋ$_{31}$で８５．３％、圧電歪定数ｄ$_{31}$で－２３５０ｐＣ／Ｎを、試料番号１５では、分極方向（縦方向振動モード）の電気機械結合係数ｋ$_{33}$で９７．４％、圧電歪定数ｄ$_{33}$で２８２０ｐＣ／Ｎを得た。又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数ｋ$_{31}$で８５．６％、圧電歪定数ｄ$_{31}$で－２３８０ｐＣ／Ｎをそれぞれ得た。周波数定数（ｆｃ$_{31}$＝ｆｒ・Ｌ）の値ｆｃ$_{31}$は、試料番号１３では、５０３Ｈｚ・ｍ、試料番号１４では、５０６Ｈｚ・ｍ、試料番号１５では、４３７Ｈｚ・ｍｍであった。

【００３７】試料番号１６では、図１１に示すように図３の（００１）面１１と直交して対向する２つの（０１０）面１３に金電極をスパッタ法で作製し、シリコンオイル中に浸漬し、１５０～２００℃の温度範囲を３０分周期で３回温度上昇・下降を繰り返しながら、直流電界４００Ｖ／ｍｍを印加した。素子を取り出したあと、エッチング液で該金電極を除去し、更に図３に示す二つの対向する（００１）面１１に金電極をスパッタ法で作製し、上記実施例で示した２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２

時間印加するか又は電界を印加したまま冷却する工程を実施し、誘電・圧電特性を測定した。その結果、分極方向（縦方向振動モード）の電気機械結合係数k_{33}で９７．８％、圧電歪定数d_{33}で２８７０ｐＣ／Ｎを得た。又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数k_{31}で８６．０％、圧電歪定数d_{31}で－２４５０ｐＣ／Ｎを得た。周波数定数（fc_{31}＝ｆｒ・Ｌ）の値fc_{31}は、４１５Ｈｚ・ｍであった。

【００３８】尚、図３の（００１）面と直交して対向する別の面として、図１１の（１００）面間に直流電界を印加後、２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を実施しても、同じ効果が得られることを確認している。

【００３９】試料番号１７では、２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程の前に、上記の単結晶圧電体素子と同じ形状の素子に、図１１に示すように図３の（００１）面１１と直交して対向する２つの（０１０）面１３に金電極をスパッタでつけ、６０℃のシリコンオイル中でピーク値５００Ｖ／ｍｍ、周期８００ｍｓｅｃのバイポーラ三角波電界を１０分間印加した。バイポーラ三角波の波形を図１３に示した。素子を取り出したあと、エッチング液で該金電極を除去し、更に図３に示す二つの対向する（００１）面１１に金電極をスパッタ法で作製し、上記実施例で示した２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を実施し、誘電・圧電特性を測定した。その結果、分極方向（縦方向振動モード）の電気機械結合係数k_{33}で９７．１％、圧電歪定数d_{33}で２７８０ｐＣ／Ｎを得た。又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数k_{31}で１８．３％、圧電歪定数d_{31}で－２３０ｐＣ／Ｎを得た。周波数定数（fc_{31}＝ｆｒ・Ｌ）の値fc_{31}は、８６３Ｈｚ・ｍであった。

【００４０】試料番号１８，１９，２０では、２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程の前に、上記の単結晶圧電体素子と同じ形状の素子を、それぞれシリコンオイル中に浸漬し、５０～９０℃、１５０～２００℃、２００～４００℃の温度範囲を５分周期で３回温度上昇・下降を繰り返した。その後、図３に示す二つの対向する（００１）面１１に金電極をスパッタ法で作製し、上記実施例で示した２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を実施し、誘電・圧電特性を測定した。

【００４１】その結果、試料番号１８では、分極方向（縦方向振動モード）の電気機械結合係数k_{33}で９７．０％、圧電歪定数d_{33}で２７６０ｐＣ／Ｎを、又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数k_{31}で１８．６％、圧電歪定数d_{31}で－２６０ｐＣ／Ｎを得た。試料番号１９では、分極方向（縦方向振動モード）の電気機械結合係数k_{33}で９７．３％、圧電歪定数d_{33}で２８１０ｐＣ／Ｎを、又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数k_{31}で１７．８％、圧電歪定数d_{31}で－１９０ｐＣ／Ｎを得た。

【００４２】試料番号２０では、分極方向（縦方向振動モード）の電気機械結合係数k_{33}で９７．２％、圧電歪定数d_{33}で２７９０ｐＣ／Ｎを、又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数k_{31}で１８．２％、圧電歪定数d_{31}で－２２０ｐＣ／Ｎをそれぞれ得た。周波数定数（fc_{31}＝ｆｒ・Ｌ）の値fc_{31}は、試料番号１８では、８３６Ｈｚ・ｍ、試料番号１９では、８９２Ｈｚ・ｍ、試料番号２０では、８４７Ｈｚ・ｍであった。

【００４３】試料番号２１では、図１１に示すように図３の（００１）面１１と直交して対向する２つの（０１０）面１３に金電極をスパッタ法で作製し、シリコンオイル中に浸漬し、１５０～２００℃の温度範囲を５分周期で３回温度上昇・下降を繰り返しながら、直流電界４００Ｖ／ｍｍを印加した。素子を取り出したあと、エッチング液で該金電極を除去し、更に図３に示す二つの対向する（００１）面１１に金電極をスパッタ法で作製し、上記実施例で示した２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程を実施し、誘電・圧電特性を測定した。その結果、分極方向（縦方向振動モード）の電気機械結合係数k_{33}で９７．７％、圧電歪定数d_{33}で２８５０ｐＣ／Ｎを得た。又、分極方向に直交する方向（横方向振動モード）の電気機械結合係数k_{31}で１７．６％、圧電歪定数d_{31}で－１５０ｐＣ／Ｎを得た。周波数定数（fc_{31}＝ｆｒ・Ｌ）の値fc_{31}は、９２４Ｈｚ・ｍであった。

【００４４】上記ドメイン制御圧電単結晶素子に際し、最終的な分極を行う工程、即ち、２０℃～２００℃の温度範囲で４００Ｖ／ｍｍ～１５００Ｖ／ｍｍの直流電界を最大２時間印加するか又は電界を印加したまま冷却する工程の前段に、分極方向と直交する方向に電界を印加し、分極方向と直交する方向の強誘電体ドメインの整列状態を制御する方法、圧電単結晶材料の低温相である菱面晶と中温相である正方晶の転移温度を挟んで加熱冷却する工程（１）、又は圧電単結晶材料の正方晶と強誘電性・圧電性の消失温度であるキュリー温度（この温度より高温では、該圧電体単結晶材料は立方晶（高温相）となる）を挟んで加熱冷却する工程（２）、又は、高温相内で加熱冷却する工程（３）、又は、工程（１）、

（２）、（３）を適宜組み合わせた工程（４）により分極方向と直交する方向の強誘電体ドメインの整列状態を制御する方法、単結晶圧電素子の分極方向に直交する方向に電界を印加する工程と、単結晶圧電素子を加熱冷却する工程とを併用する工程により分極方向と直交する方向の強誘電体ドメインの整列状態を制御する方法を実施することは、結晶育成時の徐冷過程で生成される結晶中の複数のドメインを、上記の各工程を実施することにより、より人的に制御されたものとし、上記ドメイン制御圧電単結晶素子を製造するドメイン制御単結晶圧電素子の最終的な分極を行う工程での分極方向に直交する方向のドメイン構造を、より容易に制御するために有効であること、又、本発明の第１の発明、第２の発明でいう誘電・圧電特性を向上させるために有効であることがわかった。

【００４５】

【発明の効果】本発明のドメイン制御圧電体単結晶素子及びその製造方法は、以上のように構成されているので、k_{33}が８０％以上で同時にd_{33}が８００ｐＣ／Ｎ以上、且っk_{31}が７０％以上で同時に$-d_{31}$が１２００ｐＣ／Ｎ（定義上d_{31}は、負の値を持つ）以上にした場合は、k_{31}を有効に利用した圧電単結晶素子の製造が可能であり、k_{33}が８０％以上で同時にd_{33}が８００ｐＣ／Ｎ以上、且つk_{31}が３０％以下で同時に$-d_{31}$が３００ｐＣ／Ｎ（定義上d_{31}は、負の値を持つ）以下にした場合には、k_{33}の振動モードの使用帯域内にスプリアス等の発生がないためk_{33}モードを更に効率よく利用でき、より高性能の縦方向（k_{33}）振動モード利用の圧電単結晶素子を得ることが可能となった。

【００４６】

【表１】

試料番号	分極条件 温度 ℃	電界E V/mm	時間 min	誘電・圧電特性 k_{33} %	d_{33} 10^{-12} C/N	k_{31} %	d_{31} 10^{-12} C/N	fc_{31} Hz・m	適用される請求項等
1	20	1800	10	95.6	2550	61.5	−970	701	従来例
2	60	400	150	95.3	2500	48.7	−694	773	従来例
3	100	300	120	94.0	2360	35.0	−520	755	従来例
4	40	250	10	56.0	165	18.9	−224	741	分極不十分
5	40	500	10	84.0	1190	76.0	−1310	601	請求項１
6	40	700	10	87.0	1420	77.1	−1324	603	請求項１
7	40	1000	10	95.3	2500	80.8	−1701	522	請求項１
8	40	1600	10	95.3	2500	60.9	−939	700	請求項１
9	200	400	電界冷却	80.2	960	74.7	−1263	609	請求項１
10	60	400	120	96.9	2740	26.3	−288	981	請求項２
11	20	1500	10	94.6	2300	27.1	−291	1004	請求項２
文献値１				94	2300	53	−1100	-	
文献値２				90	1734	49	−962	680-733 (平均値：707)	

【００４７】

【表２】

試料番号	処理内容	条件	誘電・圧電特性 k_{33} %	d_{33} 10^{-12} C/N	k_{31} %	d_{31} 10^{-12} C/N	fc_{31} Hz・m	対応する請求項
12	請求項５	直流電界 1000V/mm、40℃、10分	97.3	2810	85.5	−2380	483	請求項１
13	請求項６	50〜90℃、30分周期、繰り返し３回	97.5	2840	85.3	−2360	503	請求項１
14	請求項６	150〜200℃、30分周期、繰り返し３回	97.8	2880	85.3	−2350	506	請求項１
15	請求項６	200〜400℃、30分周期、繰り返し３回	97.4	2820	85.6	−2380	437	請求項１
16	請求項７	直流電界 400V/mm、150〜200℃、30分周期、繰り返し３回	97.8	2870	86.0	−2450	415	請求項１
17	請求項５	バイポーラ三角波 500V/mm、周期 800ms、10分	97.1	2780	18.3	−230	863	請求項２
18	請求項６	50〜90℃、５分周期、繰り返し３回	97.0	2760	18.6	−260	836	請求項２
19	請求項６	150〜200℃、５分周期、繰り返し３回	97.3	2810	17.8	−190	892	請求項２
20	請求項６	200〜400℃、５分周期、繰り返し３回	97.2	2790	18.2	−220	847	請求項２
21	請求項７	直流電界 400V/mm、150〜200℃、５分周期、繰り返し３回	97.7	2850	17.6	−150	924	請求項２

【図面の簡単な説明】

【図１】結晶構造の模式的斜視図である。

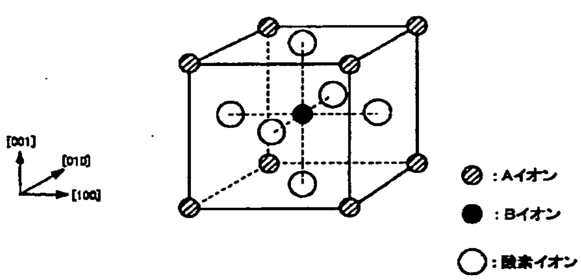
【図２】ＰＺＮ－ＰＴ（ＰＺＮＴ）の相図である。

【図３】電界印加の説明図である。

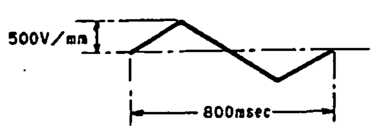
【図４】k_{31}振動モードのインピーダンスカーブの図である。

【図５】k_{31}振動モードのインピーダンスカーブの図である。
【図６】k_{31}振動モードのインピーダンスカーブの図である。
【図７】k_{31}振動モードのインピーダンスカーブの図である。
【図８】k_{31}振動モードのインピーダンスカーブの図である。
【図９】電界印加後の分極方向（厚み方向）面内のドメインの状態の図である。
【図１０】電気機械結合係数k_{31}とk_{31}振動モードの共振周波数（ｆｒ）と素子の振動方向の長さ（Ｌ）の積である周波数定数（fc_{31}＝ｆｒ・Ｌ）の値のグラフである。
【図１１】電界印加の説明図である。
【図１２】k_{33}対d_{33}のグラフである。
【図１３】バイポーラ三角波パルスの波形図である。
【符号の説明】
１０　圧電単結晶素子
１１　電極面（００１）
１２　電界
１３　電極面（０１０）

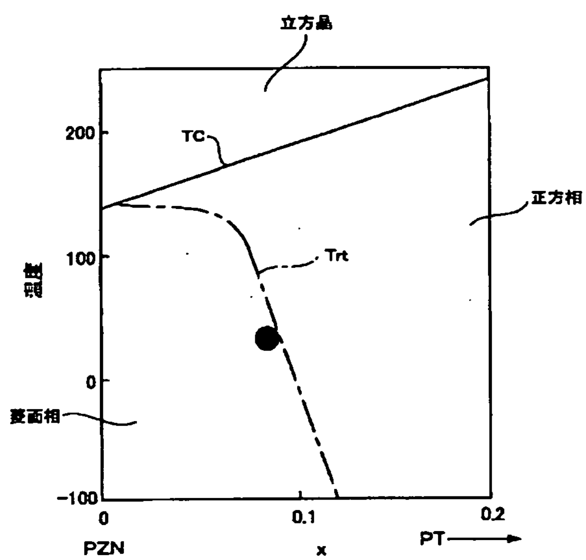
【図１】



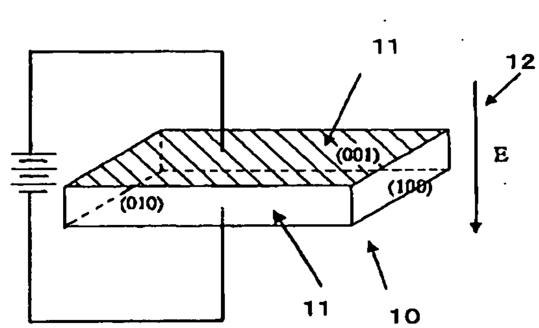
【図１３】



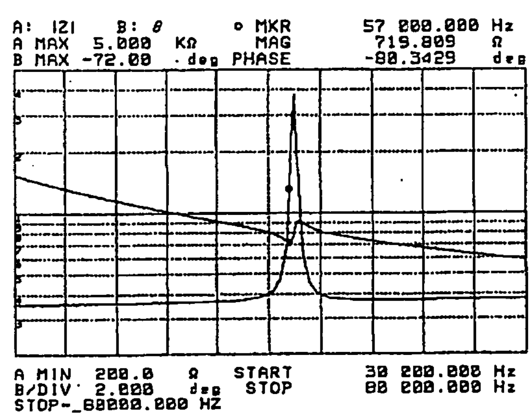
【図２】



【図３】

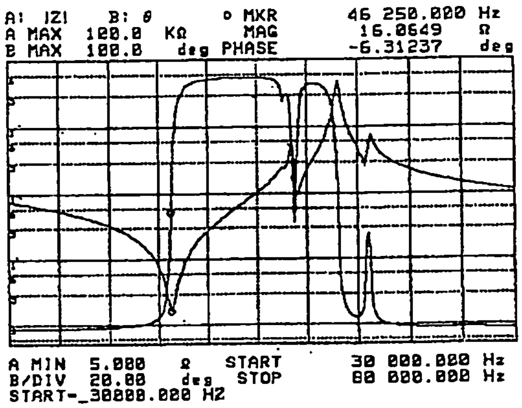


【図４】



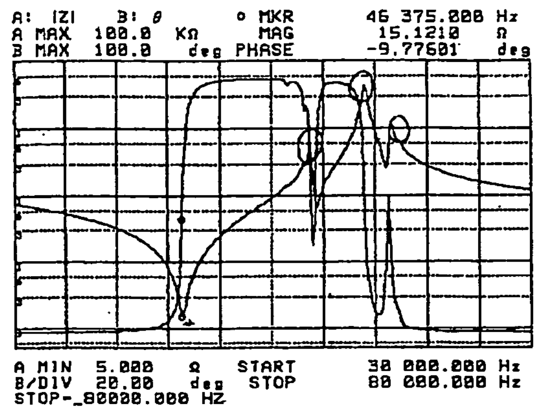
250V/mm

【図５】



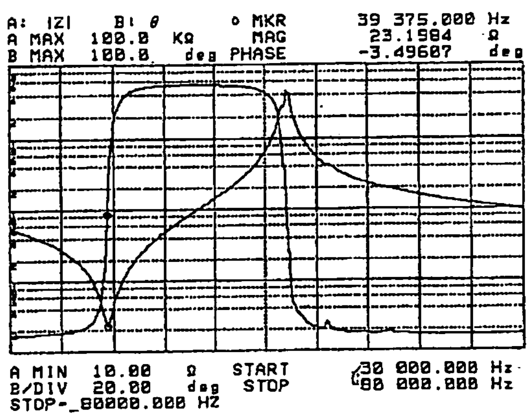
500V/mm

【図６】



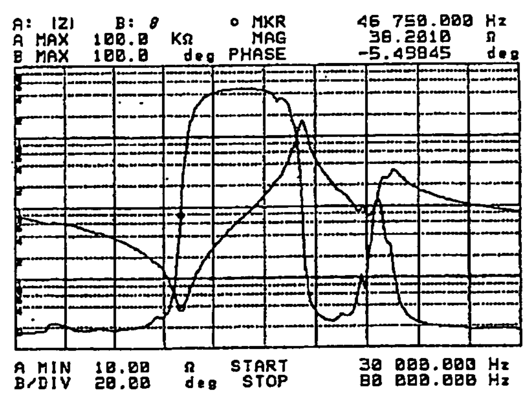
700V/mm

【図７】



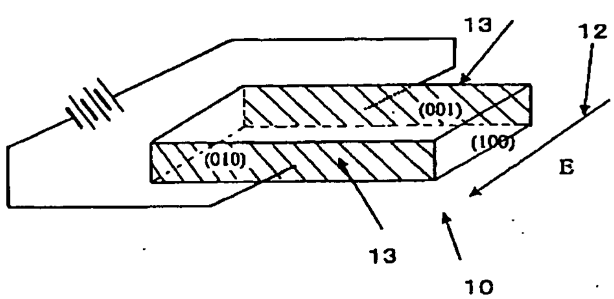
1000V/mm

【図８】

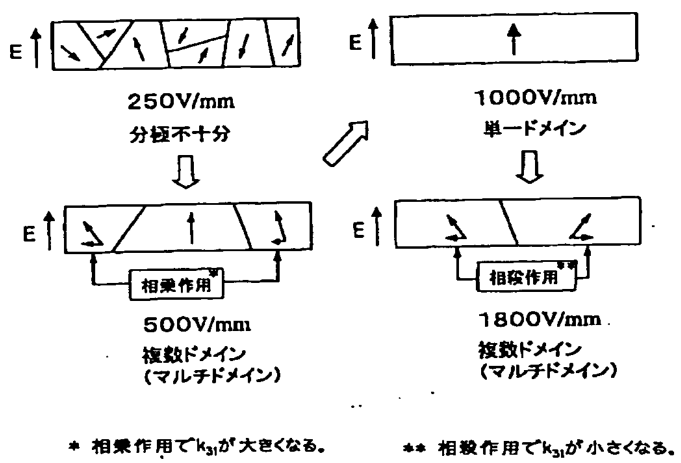


1600 V/mm

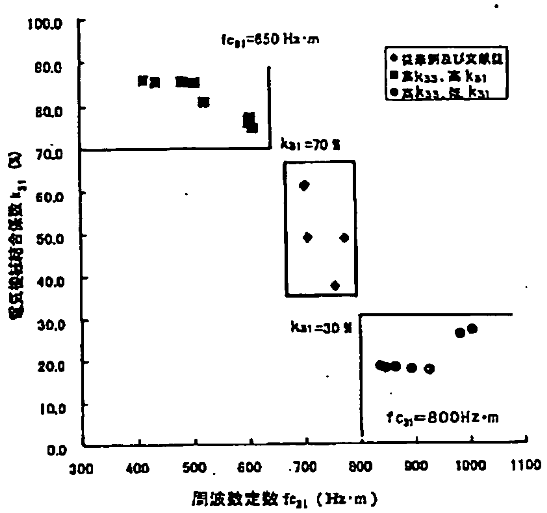
【図１１】



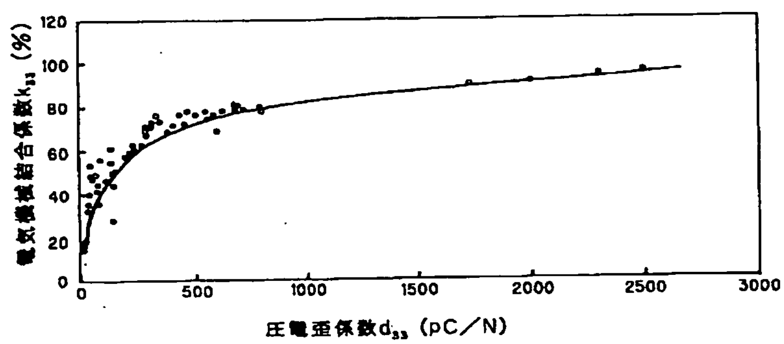
【図９】



【図１０】



【図１２】



フロントページの続き

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Ｆターム(参考) 4G031 AA03 AA07 AA14 AA15 AA16 AA17 AA18 AA19 AA23 AA26 AA27 AA32 BA10
4G077 AA02 AB06 BC43 FH02 HA11